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ORIGINAL PAPER

Acetic acid-promoted, efficient, one-pot synthesis of 2,3-dihydroquinazolin-4(1*H*)-ones

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Abstract A simple, efficient, and general method has been developed for one-pot, three-component synthesis of 2,3-dihydroquinazolin-4(1*H*)-one derivatives based on a condensation reaction strategy of isatoic anhydride, aldehydes, and amines using acetic acid under reflux conditions.

Keywords 2,3-Dihydroquinazolin-4(1*H*)-ones · Isatoic anhydride · Acetic acid · Aldehydes · Amines

Introduction

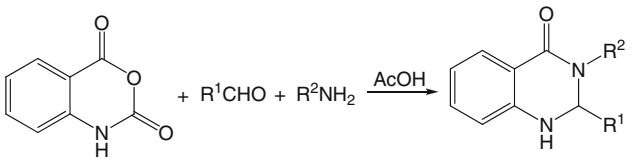
In the mainstream of current interest, multicomponent reactions (MCRs) have been proven to be a very elegant and rapid way to access complex structures in a single synthetic operation from simple building blocks, showing high atom economy and high selectivity [1–3].

2,3-Dihydroquinazolines are an important class of heterocycles with a wide range of pharmacological and biological activities [4, 5]. Additionally, these compounds can easily be oxidized to their quinazolin-4(3*H*)-one analogs, which are themselves important biologically active compounds [6, 7]. Thus, efficient synthesis of these compounds has been of great interest in recent years. A number of synthetic methods for 2,3-dihydroquinazolin-4(1*H*)-ones have been developed during the past two decades [8–10]. One-pot three-component condensation of isatoic anhydride, aldehydes, and amines is the most convenient method for preparation of these compounds. In this context, some methods and catalysts have been reported [11–22]. Developing versatile approaches towards synthesis of 2,3-dihydroquinazolin-4(1*H*)-ones still remains a highly desired goal in organic synthesis.

In view of our ongoing efforts to explore newer reactions for synthesis of heterocyclic compounds [23–25], we decided to investigate the possibility of synthesizing 2,3-dihydroquinazolin-4(1*H*)-one derivatives based on a one-pot three-component condensation reaction strategy of isatoic anhydride, aldehydes, and amines using acetic acid (Scheme 1).

Results and discussion

To optimize the reaction conditions, the reaction between benzaldehyde, aniline, and isatoic anhydride was used as a model reaction. The optimized reactant ratios were found to be 1.0 equiv. benzaldehyde, 1.1 equiv. aniline, and 1.0 equiv. isatoic anhydride in the presence of 5 cm^3 acetic acid. The expected 2,3-diphenylquinazolin-4(1*H*)-one was produced in 79% yield after 2.5 h under reflux in acetic acid. To establish the generality and scope of the acetic acid-promoted 2,3-dihydroquinazolin-4(1*H*)-one synthesis, the reaction was examined with various structurally diverse aldehydes, amines, and isatoic anhydride. The results are summarized in Table 1. The structure of the products was



Scheme 1

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Table 1 Synthesis of 2,3-dihydroquinazolin-4(1*H*)-ones

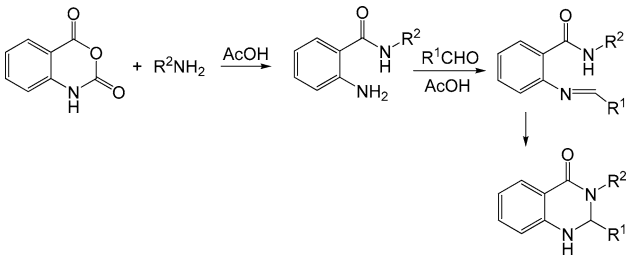
Comp.	R^1	R^2	Time (h)	Yield (%)	M.p. (°C)	Ref.
1	Ph	Ph	2.5	79	209–211	[11]
2	3-$O_2NC_6H_4$	Ph	1.5	92	186–188	[11]
3	4-$O_2NC_6H_4$	Ph	1.5	92	195–197	[11]
4	2-$O_2NC_6H_4$	Ph	1.5	91	179–181	[22]
5	4-ClC_6H_4	Ph	2	89	226–228	[11]
6	2-ClC_6H_4	Ph	2	93	136–138	[22]
7	2,6-$Cl_2C_6H_3$	Ph	1.5	93	234–236	–
8	2,4-$Cl_2C_6H_3$	Ph	1.5	91	189–191	–
9	2-Cl-6-FC_6H_3	Ph	1.5	82	208–210	–
10	Ph	$PhCH_2$	1	85	160–162	[11]
11	3-$O_2NC_6H_4$	$PhCH_2$	2	88	153–155	–
12	2-$O_2NC_6H_4$	$PhCH_2$	1	85	133–135	–
13	2-ClC_6H_4	$PhCH_2$	1.5	88	174–176	–
14	2,3-$Cl_2C_6H_3$	$PhCH_2$	1.5	88	206–208	–
15	2-Cl-6-FC_6H_3	$PhCH_2$	1	86	213–215	–
16	Ph	2-$ClC_6H_4CH_2$	2	91	175–177	–
17	3-$O_2NC_6H_4$	2-$ClC_6H_4CH_2$	2.5	89	174–176	–
18	4-$O_2NC_6H_4$	2-$ClC_6H_4CH_2$	1	90	205–207	–
19	2-ClC_6H_4	2-$ClPhCH_2$	1	93	174–176	–
20	2,6-$Cl_2C_6H_3$	2-$ClC_6H_4CH_2$	1	91	184–186	–
21	2,4-$Cl_2C_6H_3$	2-$ClC_6H_4CH_2$	1	97	141–143	–
22	2-Cl-6-FC_6H_3	2-$ClC_6H_4CH_2$	1	82	203–205	–

deduced from their infrared (IR), ^{1}H nuclear magnetic resonance (NMR), ^{13}C NMR, and elemental analysis.

The reaction can also proceed with several functionalities present on the aromatic amine, such as halogen and nitro groups. The reaction has also been done with various benzyl amines. In all these cases, the corresponding 2,3-dihydroquinazolines were obtained in good yields. It is important to note that, in the absence of acetic acid, the reaction yield was too low even at 120 °C after 5 h.

The mechanism of this reaction is believed to proceed through condensation of isatoic anhydride with amine followed by decarboxylation to yield the corresponding 2-aminobenzamide. Condensation of the aldehyde with the amino group of 2-aminobenzamide then gives an imine which undergoes cyclization to afford the 2,3-dihydroquinazoline product as shown in Scheme 2 [11].

To show the merit of the present work in comparison with results reported in the literature, we compared results of acetic acid with montmorillonite K-10 [12], silica sulfuric acid [14], $KAl(SO_4)_2{\cdot}12H_2O$ (alum) [11], $Zn(PFO)_2$ [16], $Ga(OTf)_3$ [13], and copolymer-PTSA (para-toluenesulfonic acid) [18] as catalysts in the reaction of isatoic anhydride, aniline, and benzaldehyde in the synthesis of 2,3-dihydro-2-phenylquinazolin-4(1*H*)-one. As shown in Table 2, acetic acid can act as an effective catalyst with



Scheme 2

respect to reaction times and yields of the obtained products. Thus, the present protocol with acetic acid as a catalyst is an alternate route for production of 2,3-dihydroquinazolin-4(1*H*)-ones.

In conclusion, this paper describes a convenient and efficient process for synthesis of 2,3-dihydroquinazolin-4(1*H*)-ones by one-pot reaction of isatoic anhydride, aldehydes, and amines using acetic acid under reflux conditions. Several derivatives of the title compounds with different substituents were synthesized to show the diversity of the method. The method offers several advantages, such as the lack of any toxic solvent or catalyst, simplicity of performance, low cost, and improved yields. Starting materials are inexpensive and commercially available.

Experimental

Products were characterized by comparison of their physical and spectral data with those of authentic samples. All yields refer to isolated products after recrystallization. IR and NMR spectra were recorded on PerkinElmer 781 and Bruker DPX400 machines. The progress of the reactions was monitored by thin-layer chromatography (TLC).

General procedure for preparation of 2,3-dihydroquinazolin-4(1H)-ones

A mixture of aldehyde (1 mmol), amine (1.1 mmol), and isatoic anhydride (1 mmol) in 5 cm^3 acetic acid was stirred under reflux for the appropriate time indicated in Table 1. After completion of the reaction, as indicated by TLC (ethyl acetate/*n*-hexane, 1/2), the reaction mixture was poured into 20 cm^3 ice water. On solidification, it was filtered, washed with ice water, and recrystallized from ethanol to give the pure 2,3-dihydroquinazolin-4(1*H*)-ones.

2-(2,6-Dichlorophenyl)-2,3-dihydro-3-phenylquinazolin-4(1H)-one (**7**, $C_{20}H_{14}Cl_2N_2O$)

^{1}H NMR ($CDCl_3$): δ = 4.43 (br, 1H, NH), 6.65 (d, J = 8.0 Hz, 1H, CH), 6.88–6.92 (m, 1H, Ar–H), 7.09–7.27 (m, 6H, Ar–H), 7.29–7.43 (m, 4H, Ar–H), 8.02–8.05 (m, 1H, Ar–H) ppm; ^{13}C NMR ($CDCl_3$): δ = 70.68, 114.05,

Table 2 Comparison of efficiency of various catalysts in the reaction of isatoic anhydride, aniline, and benzaldehyde

Entry	Catalyst	Conditions	Time (h)	Yield (%)	Ref.
1	Montmorillonite K-10 (0.3 g)	EtOH, reflux	6.5	80	[12]
2	Silica sulfuric acid, 20 mol% (0.06 g)	Solvent-free, 80 °C	5	80	[14]
3	$KAl(SO_4)_2{\cdot}12H_2O$ (alum, 0.2 g)	EtOH, reflux	4	78	[11]
4	$[Zn(PFO)_2]$ (0.027 g, 0.03 mmol)	H_2O/EtOH (1/3), reflux	6	82	[16]
5	$Ga(OTf)_3$ (1 mol%)	EtOH, reflux	1	79	[13]
6	Copolymer-PTSA (0.3 g)	EtOH, reflux	6.5	82	[18]
7	Acetic acid	Reflux	2.5	79	–

115.03, 118.95, 127.30, 127.53, 128.66, 129.00, 129.56, 130.51, 132.53, 133.92, 135.99, 138.69, 145.79, 163.24 ppm.

2-(2,4-Dichlorophenyl)-2,3-dihydro-3-phenylquinazolin-4(1H)-one (**8**, $C_{20}H_{14}Cl_2N_2O$)
^{1}H NMR ($CDCl_3$): $\delta = 5.21$ (br, 1H, NH), 6.42 (s, 1H, CH), 6.62 (d, J = 8.0 Hz, 1H, Ar–H), 6.89–6.94 (m, 1H, Ar–H), 7.21–7.29 (m, 3H, Ar–H), 7.31–7.40 (m, 3H, Ar–H), 7.40–7.43 (m, 1H, Ar–H), 7.52–7.55 (m, 1H, Ar–H), 7.90 (d, J = 8.0 Hz, 1H, Ar–H), 8.05 (d, J = 1.2 Hz, 1H, Ar–H) ppm; ^{13}C NMR ($CDCl_3$): $\delta = 70.76$, 114.95, 116.56, 119.90, 125.71, 126.89, 127.59, 129.06, 129.11, 130.17, 132.19, 134.19, 135.41, 135.48, 140.57, 144.46, 163.10 ppm.

2-(2-Chloro-6-fluorophenyl)-2,3-dihydro-3-phenylquinazolin-4(1H)-one (**9**, $C_{20}H_{14}ClFN_2O$)
^{1}H NMR ($CDCl_3$): $\delta = 4.65$ (br, 1H, NH), 6.67 (d, J = 8.0 Hz, 1H, CH), 6.91–6.96 (m, 2H, Ar–H), 7.11–7.14 (m, 1H, Ar–H), 7.17–7.25 (m, 3H, Ar–H), 7.28 (d, J = 2.8 Hz, 2H, Ar–H), 7.29 (d, J = 3.6 Hz, 2H, Ar–H), 7.31–7.37 (m, 1H, Ar–H), 8.05–8.08 (m, 1H, Ar–H) ppm; ^{13}C NMR ($CDCl_3$): $\delta = 69.47$, 114.49, 115.54, 115.77, 119.60, 126.06, 126.09, 127.10, 127.38, 128.98, 130.78, 130.88, 133.63, 134.13, 139.55, 145.37, 160.51, 163.03 ppm.

3-Benzyl-2,3-dihydro-2-(3-nitrophenyl)quinazolin-4(1H)-one (**11**, $C_{21}H_{17}N_3O_3$)
^{1}H NMR ($CDCl_3$): $\delta = 3.86$ (d, J = 15.6 Hz, 1H, CH_2), 5.57 (d, J = 15.6 Hz, 1H, CH_2), 5.76 (s, 1H, CH), 6.60 (d, J = 8.0 Hz, 1H, Ar–H), 6.92–6.96 (m, 1H, Ar–H), 7.23–7.27 (m, 2H, Ar–H), 7.28–7.35 (m, 4H, Ar–H), 7.48–7.52 (m, 1H, Ar–H), 7.67 (d, J = 8.0 Hz, 1H, Ar–H), 8.05–8.18 (m, 3H, Ar–H) ppm; ^{13}C NMR ($CDCl_3$): $\delta = 47.52$, 70.09, 114.88, 115.81, 119.86, 121.83, 124.02, 127.77, 127.92, 128.81, 128.84, 130.04, 132.23, 134.04, 136.21, 141.74, 144.50, 148.36, 163.09 ppm.

3-Benzyl-2,3-dihydro-2-(2-nitrophenyl)quinazolin-4(1H)-one (**12**, $C_{21}H_{17}N_3O_3$)
^{1}H NMR ($CDCl_3$): $\delta = 3.72$ (d, $J = 15.2$ Hz, 1H, CH_2), 5.32 (br, 1H, NH), 5.61 (d, $J = 15.2$ Hz, 1H, CH_2), 6.21 (s, 1H, CH), 6.51 (d, $J = 8.0$ Hz, 1H, Ar–H), 6.86–6.89 (m, 1H, Ar–H), 7.25–7.32 (m, 6H, Ar–H), 7.55–7.56 (m, 2H, Ar–H), 7.58–7.60 (m, 1H, Ar–H), 8.04–8.07 (m, 2H, Ar–H) ppm; ^{13}C NMR ($CDCl_3$): $\delta = 48.06$, 66.25, 114.76, 114.98, 119.31, 125.93, 127.91, 127.95, 128.06, 128.68, 128.86, 129.81, 134.06, 134.37, 134.77, 135.79, 143.96, 147.78, 163.73 ppm.

3-Benzyl-2-(2-chlorophenyl)-2,3-dihydroquinazolin-4(1H)-one (**13**, $C_{21}H_{17}ClN_2O$)
^{1}H NMR ($CDCl_3$): $\delta = 3.69$ (d, J = 15.2 Hz, 1H, CH_2), 4.98 (br, 1H, NH), 5.72 (d, J = 15.2 Hz, 1H, CH_2), 6.03 (s, 1H, CH), 6.51 (d, J = 8.0 Hz, 1H, Ar–H), 6.85–6.89 (m, 1H, Ar–H), 7.21–7.41 (m, 10H, Ar–H), 8.04 (dd, J = 1.2 Hz, J = 7.8 Hz, 1H, Ar–H) ppm; ^{13}C NMR ($CDCl_3$): $\delta = 47.64$, 67.27, 114.63, 115.61, 119.30, 127.34, 127.42, 127.70, 128.08, 128.69, 128.76, 130.13, 130.32, 132.11, 133.76, 136.07, 136.50, 144.54, 163.65 ppm.

3-Benzyl-2-(2,3-dichlorophenyl)-2,3-dihydroquinazolin-4(1H)-one (**14**, $C_{21}H_{16}Cl_2N_2O$)
^{1}H NMR ($CDCl_3$): $\delta = 3.67$ (d, J = 15.2 Hz, 1H, CH_2), 5.03 (br, 1H, NH), 5.72 (d, J = 15.2 Hz, 1H, CH_2), 6.03 (s, 1H, CH), 6.51 (d, J = 8.0 Hz, 1H, Ar–H), 6.85–6.89 (m, 1H, Ar–H), 7.13–7.46 (m, 9H, Ar–H), 8.03–8.05 (m, 1H, Ar–H) ppm; ^{13}C NMR ($CDCl_3$): $\delta = 47.71$, 67.64, 114.74, 115.47, 119.46, 125.43, 127.83, 127.84, 128.12, 128.70, 128.82, 130.28, 130.83, 133.90, 134.12, 136.26, 138.47, 144.27, 163.50 ppm.

3-Benzyl-2-(2-chloro-6-fluorophenyl)-2,3-dihydroquinazolin-4(1H)-one (**15**, $C_{21}H_{16}ClFN_2O$)
^{1}H NMR ($CDCl_3$): $\delta = 3.84$ (d, J = 15.2 Hz, 1H, CH_2), 4.43 (br, 1H, NH), 5.50 (d, J = 15.2 Hz, 1H, CH_2), 6.40 (s, 1H, CH), 6.85–6.89 (m, 1H, Ar–H), 6.91–6.96 (m, 1H, Ar–H), 7.20–7.29 (m, 9H, Ar–H), 8.05 (dd, J = 1.6 Hz, J = 1.6 Hz, 1H, Ar–H) ppm; ^{13}C NMR ($CDCl_3$): $\delta = 47.13$, 66.39, 113.74, 115.79, 116.03, 119.01, 126.08, 126.12, 127.58, 128.24, 128.56, 128.61, 130.59, 130.69, 133.36, 136.46, 144.71, 160.65, 163.05, 163.08, 163.17 ppm.

3-(2-Chlorobenzyl)-2,3-dihydro-2-phenylquinazolin-4(1H)-one (**16**, $C_{21}H_{17}ClN_2O$)
^{1}H NMR ($CDCl_3$): $\delta = 4.11$ (d, $J = 16.0$ Hz, 1H, CH_2), 5.45 (d, $J = 16.0$ Hz, 1H, CH_2), 5.72 (s, 1H, CH), 6.57 (d, $J = 8.0$ Hz, 1H, Ar–H), 6.89–6.93 (m, 1H, Ar–H), 7.20–7.29 (m, 2H, Ar–H), 7.30–7.59 (m, 8H, Ar–H), 8.05 (dd, $J = 1.6$ Hz, $J = 1.2$ Hz, 1H, Ar–H) ppm; ^{13}C NMR ($CDCl_3$): $\delta = 44.93$, 71.78, 114.51, 115.74, 119.32, 126.42, 127.11, 128.58, 128.76, 129.00, 129.15, 129.35, 129.48, 133.43, 133.77, 134.31, 139.25, 145.25, 163.40 ppm.

3-(2-Chlorobenzyl)-2,3-dihydro-2-(3-nitrophenyl)-quinazolin-4(1H)-one (**17**, $C_{21}H_{16}ClN_3O_3$)
^{1}H NMR ($CDCl_3$): $\delta = 4.27$ (d, J = 16.0 Hz, 1H, CH_2), 5.41 (d, J = 16.0 Hz, 1H, CH_2), 5.85 (s, 1H, CH), 6.62 (d, 1H, J = 8.0 Hz, Ar–H), 6.93–6.95 (m, 1H, Ar–H), 7.22–7.35 (m, 4H, Ar–H), 7.48–7.52 (m, 2H, Ar–H), 7.67–7.69 (m, 1H, Ar–H), 8.05–8.07 (m, 1H, Ar–H), 8.15–8.20 (m, 2H, Ar–H) ppm; ^{13}C NMR ($CDCl_3$): $\delta = 45.13$, 70.73, 114.98, 116.03, 120.14, 121.78, 124.10, 127.33, 128.92, 128.99, 129.56, 129.67, 130.06, 132.11, 133.43, 133.87, 134.10, 141.51, 144.37, 148.39, 163.13 ppm.

3-(2-Chlorobenzyl)-2,3-dihydro-2-(4-nitrophenyl)-quinazolin-4(1H)-one (**18**, $C_{21}H_{16}ClN_3O_3$)
^{1}H NMR ($CDCl_3$): $\delta = 4.19$ (d, J = 16.0 Hz, 1H, CH_2), 5.49 (d, J = 16.0 Hz, 1H, CH_2), 5.81 (s, 1H, CH), 6.60 (d, J = 8.0 Hz, 1H, Ar–H), 6.93–6.96 (m, 1H, Ar–H), 6.97–7.28 (m, 2H, Ar–H), 7.29–7.36 (m, 2H, Ar–H), 7.48–7.52 (m, 3H, Ar–H), 8.05 (dd, J = 1.2 Hz, J = 1.6 Hz, 1H, Ar–H), 8.17 (d, J = 8.0 Hz, 2H, Ar–H) ppm; ^{13}C NMR ($CDCl_3$): $\delta = 45.18$, 70.51, 114.98, 116.02, 120.17, 124.22, 127.24, 127.35, 128.88, 129.09, 129.62, 129.69, 133.55, 133.83, 134.13, 144.20, 146.29, 148.32, 163.00 ppm.

3-(2-Chlorobenzyl)-2-(2-chlorophenyl)-2,3-dihydro-quinazolin-4(1H)-one (**19**, $C_{21}H_{16}Cl_2N_2O$)
^{1}H NMR ($CDCl_3$): $\delta = 4.03$ (d, J = 16.0 Hz, 1H, CH_2), 5.61 (d, $J = 16.0$ Hz, 1H, CH_2), 6.08 (s, 1H, CH), 6.54 (d, $J = 8.0$ Hz, 1H, Ar–H), 6.85–6.87 (m, 1H, Ar–H), 7.21–7.29 (m, 4H, Ar–H), 7.30–7.44 (m, 4H, Ar–H), 7.57 (dd, $J = 2.0$ Hz, $J = 1.6$ Hz, 1H, Ar–H), 8.03 (dd, $J = 1.6$ Hz, $J = 1.2$ Hz, 1H, Ar–H) ppm; ^{13}C NMR ($CDCl_3$): $\delta = 45.60$, 68.03, 114.73, 115.62, 119.40, 127.22, 127.29, 127.38, 128.68, 128.89, 129.40, 129.57, 130.16, 130.37, 132.12, 133.65, 133.86, 134.06, 135.92, 144.65, 163.83 ppm.

3-(2-Chlorobenzyl)-2-(2,6-dichlorophenyl)-2,3-dihydroquinazolin-4(1H)-one (**20**, $C_{21}H_{15}Cl_3N_2O$)
^{1}H NMR ($CDCl_3$): $\delta = 4.05$ (d, J = 16.0 Hz, 1H, CH_2), 4.46 (br, 1H, NH), 5.50 (d, J = 16.0 Hz, 1H, CH_2), 6.56 (d, J = 8.0 Hz, 1H, CH), 6.79 (s, 1H, Ar–H), 6.79–6.89 (m, 1H, Ar–H), 7.13–7.43 (m, 8H, Ar–H), 8.04 (dd, J = 1.6 Hz, J = 1.2 Hz, 1H, Ar–H) ppm; ^{13}C NMR ($CDCl_3$): $\delta = 43.68$, 68.61, 113.56, 114.20, 118.58, 127.04, 128.49, 128.77, 129.55, 129.44, 130.62, 132.07, 133.30, 133.58, 133.79, 133.89, 136.12, 145.61, 163.51 ppm.

3-(2-Chlorobenzyl)-2-(2,4-dichlorophenyl)-2,3-dihydroquinazolin-4(1H)-one (**21**, $C_{21}H_{15}Cl_3N_2O$)
^{1}H NMR ($CDCl_3$): $\delta = 4.05$ (d, J = 16.0 Hz, 1H, CH_2), 5.01 (br, 1H, NH), 5.57 (d, J = 16.0 Hz, 1H, CH_2), 6.04 (s, 1H, CH), 6.54 (d, J = 8.0 Hz, 1H, Ar–H), 6.87–6.91 (m, 1H, Ar–H), 7.16–7.44 (m, 7H, Ar–H), 7.56 (dd, J = 2.0 Hz, J = 7.8 Hz, 1H, Ar–H), 8.02 (dd, J = 1.6 Hz, J = 8.0 Hz, 1H, Ar–H) ppm; ^{13}C NMR ($CDCl_3$): $\delta = 45.52$, 67.63, 114.80, 115.56, 119.83, 127.33, 127.60, 128.07, 128.71, 129.02, 129.39, 129.59, 130.23, 132.78, 133.70, 133.88, 134.01, 134.65, 135.40, 144.38, 163.65 ppm.

3-(2-Chlorobenzyl)-2-(2-chloro-6-fluorophenyl)-2,3-dihydroquinazolin-4(1H)-one (**22**, $C_{21}H_{15}Cl_2FN_2O$)
^{1}H NMR ($CDCl_3$): $\delta = 4.17$ (d, $J = 16.0$ Hz, 1H, CH_2), 4.72 (br, 1H, NH), 5.49 (d, $J = 16.0$ Hz, 1H, CH_2), 6.40 (s, 1H, CH), 6.56 (d, $J = 8.0$ Hz, 1H, Ar–H), 6.87–6.98 (m, 2H, Ar–H), 7.19–7.29 (m, 4H, Ar–H), 7.30–7.49 (m, 3H, Ar–H), 8.06 (dd, $J = 1.6$ Hz, $J = 1.2$ Hz, 1H, Ar–H) ppm; ^{13}C NMR ($CDCl_3$): $\delta = 44.64$, 66.98, 113.99, 115.89, 116.13, 119.28, 126.21, 126.24, 127.16, 128.67, 128.72, 129.43, 129.51, 130.64, 130.74, 133.44, 133.66, 133.85, 144.77, 160.45, 162.97, 163.39 ppm.

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