Synthesis of 3-aryl-3,4-dihydroquinazolin-2(1*H*)-ones with the aid of low-valent titanium reagent ($TiCl_4$–Sm)

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A short and facile synthesis of a series of 3-aryl-3,4-dihydroquinazolin-2(1*H*)-ones was accomplished in good yields via the novel reductive cyclisation of 2-nitrobenzylamines with bis(trichloromethyl)carbonate (triphosgene) promoted by $TiCl_4$/Sm system. The structures of the products were characterised by IR, ^{1}H NMR and elemental analysis and the structure of **3a** was confirmed by X-ray diffraction analysis.

Keywords: 3-aryl-3,4-dihydroquinazolin-2(1*H*)-one, low-valent titanium reagent, triphosgene, 2-nitrobenzylamine

Low-valent titanium reagents have an exceedingly high ability to promote reductive coupling of carbonyl compounds and are attracting increasing interest in organic synthesis.[1] Many other functional groups can also be coupled.[2] Recently, we have reported the low-valent titanium-induced intermolecular reductive coupling reaction of carboxylic derivatives with aromatic ketones,[3] the intramolecular reductive coupling reaction of 4,4-dicyano-1,3-diarylbutan-1-one,[4] the cyclodimerisation of *α,β*-unsaturated ketones[5] and the intramolecular reductive coupling reaction of ketomalononitriles.[6]

Quinazolinone derivatives are interesting because of their diverse range of biological activities such as anticancer, diuretic, anti-inflammatory, anticonvulsant, and antihypertensive agents.[7] Recently, these compounds have also been reported as Na^+/Ca^{2+} exchanger inhibitor.[8] The main synthetic approaches to such compounds consist of the condensation of imidates with anthranilic acid,[9] the reaction of anthranilamides with aldehydes,[10] Pd-catalysed cyclisation reaction of aryl-benzyl ureas[11] and the aza-Wittig reactions of *α*–azido-substituted aromatic imides.[12] Although some of the methods provide useful strategies for the synthesis of these bicyclic compounds, they suffer major drawbacks, such as involving a multistep synthesis, producing low yields, and requiring expensive catalyst, and are, thus, less desirable commerically.

Our interest in recent years has been focused on the application of the low-valent titanium reagent in organic synthesis. We have previously reported the synthesis of bioactive molecules such as indoles,[13] 2-aminoquinolines,[14] quinazolin-4(3*H*)-ones,[15] imidazo[1,2-*c*]quinazolines[16] and pyrroles[17] with the aid of this reagent. Here we wish to describe a method induced by the $TiCl_4$/Sm system for the preparation of 3-aryl-3,4-dihydroquinazolin-2(1*H*)-ones using 2-nitrobenzylamines as the starting materials.

When 2-nitrobenzylamines **1** and triphosgene **2** were treated with low-valent titanium prepared from titanium tetrachloride and samarium powder in anhydrous THF, the reductive cyclisation products 3-aryl-3,4-dihydroquinazolin-2(1*H*)-ones **3** were obtained (Scheme 1).

Table 1 summarises our results on the intermolecular reductive cyclisation of 2-nitrobenzyl- amines and triphosgene. All substrates were cyclised in good yields to afford only one product **3**. The reductive products of nitro compounds were not detected. The chloro, bromo and fluoro groups of the substrates could not be reduced under the reaction conditions. As shown in Table 1, this protocol could be applied not only to 3-aryl substituted with electron-donating groups, but also to 3-aryl substituted with electron-withdrawing groups.

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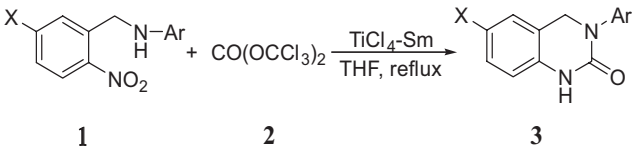

1 **2** **3**

Scheme 1

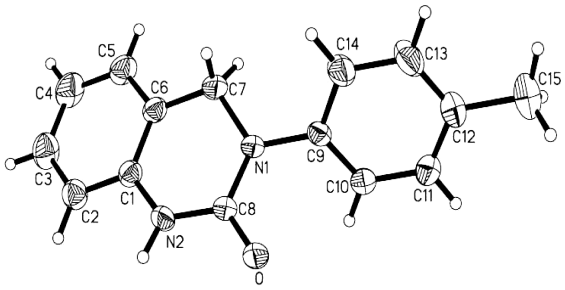

Fig. 1 Molecular structure of **3a** showing the atom numbering scheme.

Table 1 The synthesis of 3-aryl-3,4-dihydroquinazolin-2(1*H*)-ones promoted by $TiCl_4$/Sm

Entry	X	Ar	Yield/%	Mp/°C
3a	H	4-$CH_3C_6H_4$	96	222–224
3b	H	4-$CH_3OC_6H_4$	82	248–249
3c	H	4-BrC_6H_4	93	282–284
3d	H	4-FC_6H_4	95	203–205
3e	H	3-Cl-4-$CH_3C_6H_3$	94	185–187
3f	H	3-Cl-4-FC_6H_3	92	174–175
3g	H	3-$CH_3C_6H_4$	94	166–167
3h	Cl	4-$CH_3C_6H_4$	93	218–219
3i	Cl	4-BrC_6H_4	89	225–227
3j	Cl	4-ClC_6H_4	86	206–207
3k	Cl	3-Cl-4-$CH_3C_6H_3$	90	214–216
3l	Cl	3-$CH_3C_6H_4$	91	198–200
3m	Cl	3-$CH_3OC_6H_4$	94	157–159
3n	Cl	2-ClC_6H_4	89	185–187

The structures of compounds **3** were characterised by IR, ^{1}H NMR and elemental analysis. The structure of **3a** was further confirmed by X-ray diffraction[18] (Fig. 1).

In summary, a series of 3-aryl-3,4-dihydroquinazolin-2(1*H*)-ones were synthesised via reductive cyclisation of 2-nitrobenzylamines with triphosgen induced by the $TiCl_4$/Sm. The advantages of our new method are the easily accessible starting materials, convenient manipulation and high yields.

Experimental

THF was distilled from sodium-benzophenone immediately prior to use. All reactions were conducted under N_2 atmosphere. Melting points are uncorrected. IR spectra were recorded on Tensor 27 spectrometer in KBr with absorptions in cm^{-1}. ^1H NMR spectra were determined on Bruker DPX 400 MHz spectrometer in DMSO-d_6 solution. *J* values are in Hz. Chemical shifts are expressed in ppm downfield from internal standard TMS. Microanalyses were carried out on Perkin-Elmer 2400 II instruments. X-ray diffraction was recorded on a Siemens P4 diffractometer.

General procedure for the synthesis of 3-aryl-3,4-dihydroquinazolin-2(1H)-ones

$TiCl_4$ (1.1 ml, 10 mmol) was added dropwise using a syringe to a stirred suspension of Sm powder (1.5 g, 10 mmol) in freshly distilled anhydrous THF (20 ml) at r.t. under a dry N_2 atmosphere. After completion of the addition, the mixture was refluxed for 2 h. The suspension of the low-valent titanium reagent formed was cooled to r.t. and a solution of 2-nitrobenzylamines (2 mmol) and triphosgene (3 mmol) in THF (10 ml) was added dropwise. The reaction mixture was then refluxed for 0.5 h under N_2. After this period, the TLC analysis of the mixture showed the reaction to be completed. The reaction mixture was quenched with 5% HCl (50 ml) and extracted with $ClCH_2CH_2Cl$ (3 × 50 ml). The combined extracts were washed with water (3 × 50 ml) and dried over anhydrous Na_2SO_4. After evaporation of the solvent under reduced pressure, the crude product was purified by recrystallisation from 95% ethanol.

3-(4-Methylphenyl)-3,4-dihydroquinazolin-2(1H)-one (**3a**): IR: *ν*/cm^{-1} 3214, 3069, 2913, 1692, 1606, 1512, 1423, 1306, 1253, 1217, 1161, 1111, 1035, 1020, 1003, 974, 931, 859, 808, 784; ^1H NMR (DMSO-d_6): δ 2.31 (3H, s, CH_3), 4.77 (2H, s, CH_2), 6.86–6.93 (2H, m, ArH), 7.15–7.26 (6H, m, ArH), 9.53 (1H, s, NH); Anal. calcd for $C_{15}H_{14}N_2O$: C 75.61, H 5.92, N 11.76; found C 75.72, H 5.84, N 11.89.

3-(4-Methoxyphenyl)-3,4-dihydroquinazolin-2(1H)-one (**3b**): IR: *ν*/cm^{-1} 3210, 3071, 2917, 2838, 1666, 1604, 1507, 1475, 1426, 1322, 1298, 1242, 1184, 1168, 1029, 859, 844, 811, 782, 756; ^1H NMR (DMSO-d_6): δ 3.77 (3H, s, CH_3O), 4.75 (2H, s, CH_2), 6.85–6.91 (2H, m, ArH), 6.94 (2H, d, *J* = 8.8 Hz, ArH), 7.14–7.21 (2H, m, ArH), 7.28 (2H, d, *J* = 8.8 Hz, ArH), 9.49 (1H, s, NH); Anal. calcd for $C_{15}H_{14}N_2O_2$: C 70.85, H 5.55, N 11.02; found C 70.97, H 5.39, N 11.16.

3-(4-Bromophenyl)-3,4-dihydroquinazolin-2(1H)-one (**3c**): IR: *ν*/cm^{-1} 3203, 3067, 2916, 1667, 1603, 1587, 1574, 1489, 1463, 1427, 1398, 1319, 1301, 1267, 1224, 1071, 1009, 833, 798, 756, 728; ^1H NMR (DMSO-d_6): δ 4.81 (2H, s, CH_2), 6.88 (1H, d, *J* = 7.6 Hz, ArH), 6.93 (1H, t, *J* = 7.2 Hz, ArH), 7.16–7.21 (2H, m, ArH), 7.36 (2H, d, *J* = 8.4 Hz, ArH), 7.56 (2H, d, *J* = 8.4 Hz, ArH), 9.68 (1H, s, NH); Anal. calcd for $C_{14}H_{11}BrN_2O$: C 55.47, H 3.66, N 9.24; found C 55.54, H 3.63, N 9.18.

3-(4-Fluorophenyl)-3,4-dihydroquinazolin-2(1H)-one (**3d**): IR: *ν*/cm^{-1} 3207, 3066, 2911, 1670, 1598, 1509, 1470, 1440, 1426, 1411, 1320, 1296, 1263, 1215, 1155, 840, 793, 750; ^1H NMR (DMSO-d_6): δ 4.80 (2H, s, CH_2), 6.87 (1H, d, *J* = 8.0 Hz, ArH), 6.91–6.94 (1H, m, ArH), 7.15–7.24 (4H, m, ArH), 7.40–7.43 (2H, m, ArH), 9.60 (1H, s, NH); Anal. calcd for $C_{14}H_{11}FN_2O$: C 69.41, H 4.58, N 11.56; found C 69.57, H 4.53, N 11.64.

3-(3-Chloro-4-methylphenyl)-3,4-dihydroquinazolin-2(1H)-one (**3e**): IR: *ν*/cm^{-1} 3211, 3064, 2992, 2918, 1672, 1604, 1566, 1485, 1473, 1419, 1382, 1307, 1261, 1216, 1161, 1045, 1016, 994, 863, 817, 735, 664; ^1H NMR (DMSO-d_6): δ 2.33 (3H, s, CH_3), 4.81 (2H, s, CH_2), 6.88 (1H, d, *J* = 7.6 Hz, ArH), 6.91–6.95 (1H, m, ArH), 7.16–7.21 (2H, m, ArH), 7.27 (1H, d, *J* = 8.0 Hz, ArH), 7.35 (1H, d, *J* = 8.4 Hz, ArH), 7.47 (1H, s, ArH), 9.64 (1H, s, NH); Anal. calcd for $C_{15}H_{13}ClN_2O$: C 66.06, H 4.80, N 10.27; found C 66.19, H 4.85, N 10.12.

3-(3-Chloro-4-fluorophenyl)-3,4-dihydroquinazolin-2(1H)-one (**3f**): IR: *ν*/cm^{-1} 3206, 3068, 2990, 2921, 1694, 1604, 1505, 1470, 1445, 1425, 1404, 1312, 1257, 1240, 1218, 1157, 1059, 1014, 857, 810, 794, 738, 716; ^1H NMR (DMSO-d_6): δ 4.83 (2H, s, CH_2), 6.88 (1H, d, *J* = 8.0 Hz, ArH), 6.92–6.96 (1H, m, ArH), 7.16–7.26 (2H, m, ArH), 7.40–7.46 (2H, m, ArH), 7.65–7.67 (1H, m, ArH), 9.69 (1H, s, NH); Anal. calcd for $C_{14}H_{10}ClFN_2O$: C 60.77, H 3.64, N 10.12; found C 60.83, H 3.58, N 10.20.

3-(3-Methylphenyl)-3,4-dihydroquinazolin-2(1H)-one (**3g**): IR: *ν*/cm^{-1} 3200, 3060, 2916, 1675, 1602, 1475, 1441, 1423, 1321, 1299, 1273, 1214, 1161, 1088, 1035, 1002, 859, 808, 780, 759, 740, 702; ^1H NMR (DMSO-d_6): δ 2.32 (3H, s, CH_3), 4.79 (2H, s, CH_2), 6.87–6.95 (2H, m, ArH), 7.03 (1H, d, *J* = 7.2 Hz, ArH), 7.15–7.29 (5H, m, ArH), 9.55 (1H, s, NH),; Anal. calcd for $C_{15}H_{14}N_2O$: C 75.61, H 5.92, N 11.76; found C 75.54, H 5.87, N 11.91.

6-Chloro-3-(4-methylphenyl)-3,4-dihydroquinazolin-2(1H)-one (**3h**): IR: *ν*/cm^{-1} 3195, 3045, 2924, 1670, 1599, 1492, 1467, 1444, 1399, 1304, 1275, 1205, 1084, 927, 878, 819, 747, 718, 668; ^1H NMR (DMSO-d_6): δ 2.31 (3H, s, CH_3), 4.78 (2H, s, CH_2), 6.87 (1H, d, *J* = 8.4 Hz, ArH), 7.17–7.20 (2H, m, ArH), 7.23–7.24 (2H, m, ArH), 7.25–7.28 (2H, m, ArH), 9.66 (1H, s, NH); Anal. calcd for $C_{15}H_{13}ClN_2O$: C 66.06, H 4.80, N 10.27; found C 66.19, H 4.74, N 10.41.

6-Chloro-3-(4-bromophenyl)-3,4-dihydroquinazolin-2(1H)-one (**3i**): IR: *ν*/cm^{-1} 3203, 3087, 2936, 1687, 1600, 1492, 1467, 1438, 1391, 1297, 1272, 1218, 1204, 1072, 1008, 927, 826, 806, 732, 709; ^1H NMR (DMSO-d_6): δ 4.82 (2H, s, CH_2), 6.88 (1H, d, *J* = 8.4 Hz, ArH), 7.25–7.28 (2H, m, ArH), 7.35 (2H, d, *J* = 8.4 Hz, ArH), 7.57 (2H, d, *J* = 8.4 Hz, ArH), 9.79 (1H, s, NH); Anal. calcd for $C_{14}H_{10}BrClN_2O$: C 49.81, H 2.99, N 8.30; found C 49.93, H 2.95, N 8.38.

6-Chloro-3-(4-chlorophenyl)-3,4-dihydroquinazolin-2(1H)-one (**3j**): IR: *ν*/cm^{-1} 3222, 3095, 2938, 1674, 1593, 1489, 1457, 1442, 1394, 1307, 1273, 1211, 1165, 1093, 1015, 927, 853, 824, 748, 716; ^1H NMR (DMSO-d_6): δ 4.82 (2H, s, CH_2), 6.88 (1H, d, *J* = 8.4 Hz, ArH), 7.24–7.28 (2H, m, ArH), 7.40 (2H, d, *J* = 8.4 Hz, ArH), 7.42 (2H, d, *J* = 8.4 Hz, ArH), 9.79 (1H, s, NH); Anal. calcd for $C_{14}H_{10}Cl_2N_2O$: C 57.36, H 3.44, N 9.56; found C 57.48, H 3.39, N 9.63.

6-Chloro-3-(3-chloro-4-methylphenyl)-3,4-dihydroquinazolin-2(1H)-one (**3k**): IR: *ν*/cm^{-1} 3200, 3057, 2930, 1675, 1598, 1506, 1463, 1389, 1304, 1209, 1160, 1081, 1047, 927, 879, 825, 781, 747, 703, 667; ^1H NMR (DMSO-d_6): δ 2.32 (3H, s, CH_3), 4.80 (2H, s, CH_2), 6.87 (1H, d, *J* = 8.0 Hz, ArH), 7.22–7.28 (3H, m, ArH), 7.34 (1H, d, *J* = 7.6 Hz, ArH), 7.46 (1H, s, ArH), 9.78 (1H, s, NH); Anal. calcd for $C_{15}H_{12}Cl_2N_2O$: C 58.65, H 3.94, N 9.12; found C 58.61, H 3.89, N 9.27.

6-Chloro-3-(3-methylphenyl)-3,4-dihydroquinazolin-2(1H)-one (**3l**): IR: *ν*/cm^{-1} 3204, 3049, 2916, 1684, 1602, 1473, 1430, 1394, 1299, 1272, 1207, 1162, 1082, 1030, 927, 870, 814, 779, 699; ^1H NMR (DMSO-d_6): δ 2.35 (3H, s, CH_3), 4.79 (2H, s, CH_2), 6.88 (1H, d, *J* = 8.4 Hz, ArH), 7.04 (1H, d, *J* = 7.6 Hz, ArH), 7.12–7.19 (2H, m, ArH), 7.23–7.29 (3H, m, ArH), 9.69 (1H, s, NH); Anal. calcd for $C_{15}H_{13}ClN_2O$: C 66.06, H 4.80, N 10.27; found C 66.14, H 4.76, N 10.35.

6-Chloro-3-(3-methoxyphenyl)-3,4-dihydroquinazolin-2(1H)-one (**3m**): IR: *ν*/cm^{-1} 3197, 3047, 2920, 1668, 1602, 1494, 1396, 1319, 1304, 1254, 1212, 1164, 1150, 1086, 1037, 1015, 922, 867, 845, 818, 743, 698; ^1H NMR (DMSO-d_6): δ 3.76 (3H, s, CH_3O), 4.81 (2H, s, CH_2), 6.81 (1H, d, *J* = 8.0 Hz, ArH), 6.88 (1H, d, *J* = 8.4 Hz, ArH), 6.93–6.95 (2H, m, ArH), 7.24–7.31 (3H, m, ArH), 9.71 (1H, s, NH); Anal. calcd for $C_{15}H_{13}ClN_2O_2$: C 62.40, H 4.54, N 9.70; found C 62.53, H 4.47, N 9.85.

6-Chloro-3-(2-chlorophenyl)-3,4-dihydroquinazolin-2(1H)-one (**3n**): IR: *ν*/cm^{-1} 3196, 3053, 2928, 1670, 1587, 1482, 1442, 1398, 1305, 1271, 1206, 1128, 1088, 1065, 927, 818, 744, 725, 668; ^1H NMR (DMSO-d_6): δ 4.61 (1H, d, *J* = 14.4 Hz, CH), 4.80 (1H, d, *J* = 14.4 Hz, CH), 6.87 (1H, d, *J* = 8.4 Hz, ArH), 7.24–7.26 (2H, m, ArH), 7.36-7.46 (2H,m, ArH), 7.54–7.59 (2H, m, ArH), 9.73 (1H, s, NH); Anal. calcd for $C_{14}H_{10}Cl_2N_2O$: C 57.36, H 3.44, N 9.56; found C 57.53, H 3.39, N 9.65.

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18 Crystal suitable for X-ray analysis was obtained by slow evaporation of an EtOH solution of **3a.** $C_{15}H_{14}N_2O$, M = 238.28, triclinic, space group P-1, $a = 6.344(1)$, $b = 9.504(1)$, $c = 10.726(2)$ Å, $\alpha = 78.92(1)°$, $\beta = 83.04(1)°$, $\gamma = 78.91(1)°$, V = 620.38(18) Å^3, Z = 2, $\rho_{calcd} = 1.276$ g·cm^{-3}, F(000) = 252, $\mu(M_oK_\alpha) = 0.082$ mm^{-1}, colourless block crystals, crystal size 0.60 mm × 0.50 mm × 0.20 mm. Intensity data were collected at 296K on a Siemens P4 diffractometer with graphite-monochromated M_oK_α radiation (l = 0.71073 Å): 2311 independent reflections were collected using *w* scan mode in the range of 1.94° to 25.49°, of which 1395 intensity data with [$I>2\sigma$ (I)] were observed. The corrections for L*p* factors were applied. The structure was solved by direct methods and expanded using Fourier techniques. Non-hydrogen atoms were refined anisotropically. Hydrogen atoms were introduced in the F value calculation but fixed during the structure refinement. A full matrix least-squares refinement gave final $R_1 = 0.0452$ and $wR_2 = 0.1146$, with $w = 1/[\sigma^2(Fo^2) + (0.0668P)^2]$ where $P = (Fo^2 + 2F_c^2)/3$, $S = 0.957$. The maximum peak in the final difference Fourier map was 0.150 e/Å^3 and the minimum peak was -0.126 e/Å^3. All calculations were performed using TEXSAN program package. Full crystallographic data has been deposited at the Cambridge Crystallographic Data Centre, deposition number CCDC 659078. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB2 1E2, UK (fax: (+ 44)1223-336-033; E-mail: deposit@ccdc.cam.ac.uk).