SHORT PAPER

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A facile synthesis of 1,2-dihydroquinazolin-4(3*H*)-ones with the aid of a low-valent titanium reagent†

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A short and facile synthesis of a series of 1,2-dihydroquiazolin-4(3*H*)-ones was accomplished in good yields via the novel reductive cyclisation of *o*-nitrobenzamides with aldehydes or ketones promoted by the $TiCl_4$/Sm system. Structures were established on the basis of elemental analysis, IR, ^{1}H NMR and confirmed by a single-crystal X-ray diffraction analysis. The advantages of our method are easily accessible starting materials, convenient manipulation and moderate to high yields.

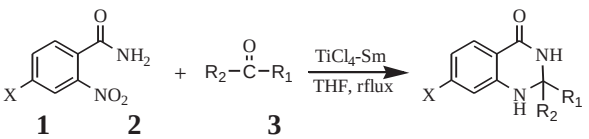
Keywords: quinazolin-4(3*H*)-one, low-valent titanium, *o*-nitrobenzamide, aldehyde, ketone

Low-valent titanium reagents have an exceedingly high ability to promote reductive coupling of carbonyl compounds and are attracting increasing interest in organic synthesis. Many other functional groups can also be coupled.[1] Recently, we have reported that the cyclodimerisation of α,β-unsaturated ketones and α,β-unsaturated nitrile compounds promoted by this reagent, yielded functional cyclopentanes[2] and cyclopentenes[3] respectively.

Quinazolin-4(3*H*)-ones are in demand because of their potential biological and pharmaceutical activities[4]. Unfortunately, synthetic methods for the elaboration of this bicyclic system are not general in scope, and involve multistep, and often low-yielding, reaction sequences. The main synthetic approaches to such compounds consist of preliminary amidation of 2-aminobenzonitrile, 2-aminobenzotic acid or ethyl 2-aminobenzate[5] and the aza-Wittig reactions of α-azido-substituted aromatic imides.[6] Different one-pot syntheses have been described, but the condensation of 2-aminobenzoic acid with amides or nitriles requires either high temperature or must be affected in a sealed tube at 200˚C.[7] Here we wish to describe a new method induced by the $TiCl_4$/Sm system for the preparation of 1,2-dihydroquinazolin-4(3*H*)-ones using *o*-nitrobenzamides as the starting material.

When *o*-nitrobenzamides **1** and aromatic aldehydes or ketones **2** were treated with low-valent titanium, prepared from titanium tetrachloride and Sm powder in anhydrous THF, the reductive cyclisation products 1,2-dihydroquinazolin-4(3*H*)-ones **3** were obtained in good yields (Scheme 1). The results are summarised in Table 1.

On the other hand, the reaction of *o*-nitrobenzamide **1** and the cyclic ketone **4** with the same reagent afforded 2,2-polymethylene-1,2- dihydroquinazolin-4(3*H*)-ones **5** (Scheme 2) and the results are summarised in Table 2. However, *o*-nitrobenzamide failed to react with acetophenone or



1 **2** **3**

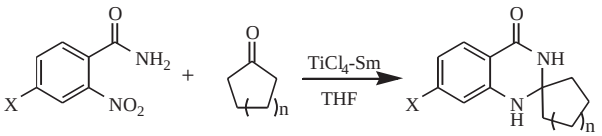
Scheme 1

* To receive any correspondence.

† This is a Short Paper, there is therefore no corresponding material in *J Chem. Research (M)*.

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

Entry	Compound	X	R_1	R_2	Isolated Yield/%
1	**3a**	H	4-ClC_6H_4	H	91
2	**3b**	H	3,4-$(OCH_3)_2C_6H_3$	H	92
3	**3c**	H	3,4-$OCH_2OC_6H_3$	H	93
4	**3d**	H	C_6H_5	H	83
5	**3e**	H	4-$CH_3C_6H_4$	H	91
6	**3f**	Cl	4-ClC_6H_4	H	82
7	**3g**	Cl	4-FC_6H_4	H	79
8	**3h**	Cl	4-$CH_3C_6H_4$	H	85
9	**3i**	Cl	4-$CH_3OC_6H_4$	H	87
10	**3j**	H	CH_3	CH_3	88
11	**3k**	H	C_2H_5	CH_3	78
12	**3l**	H	C_2H_5	C_2H_5	86
13	**3m**	Cl	CH_3	CH_3	79
14	**3n**	Cl	C_2H_5	CH_3	74
15	**3o**	Cl	C_2H_5	C_2H_5	71



1 **4** **5**

Scheme 2

Table 2 The reductive cyclisation of *o*-nitrobenzamides and cyclic ketones

Entry	Compound	X	*n*	Isolated yield/%
1	**5a**	H	1	86
2	**5b**	Cl	1	91
3	**5c**	H	2	72
4	**5d**	Cl	2	89

1-tetralone to give the same products under the same reaction conditions.

The structures of **3** and **5** were confirmed by IR, ^{1}H NMR and elemental analysis. In these structures, **5c** was further confirmed by X-ray analysis.

In summary, a series of 1,2-dihydro- quinazolin-4(3*H*)-ones were synthesized via reductive cyclisation induced by the $TiCl_4$/Sm system of *o*-nitrobenzamides with aldehydes or ketones. The advantages of our method are easily accessible

starting materials, convenient manipulation and moderate to high yields.

Experimental

Tetrahydrofuran (THF) was distilled from sodium-benzophenone immediately prior to use. All reactions were performed under a dinitrogen atmosphere. Melting points were uncorrected. ^{1}H NMR spectra were obtained for solution in $CDCl_3$ with Me_4Si as internal standard using an Inova-400 spectrometer. Microanalyses were carried out using a Perkin-Elmer 2400 II analyzer. IR spectra were recorded on an FTIR-8101 spectrometer in KBr. X-ray diffraction was recorded on a Siemens P4 diffractometer.

General procedure for the synthesis of 1, 2-Dihydroquinazolin-4(3H)-ones (3 and 5): $TiCl_4$ (1.1 ml, 10 mmol) was added dropwise using a syringe to a stirred suspension of samariam dust (1.5 g, 10 mmol) in freshly distilled anhydrous THF (15 ml) at room temperature under a dry dinitrogen atmosphere. After completion of the addition, the mixture was refluxed for 2 h. The suspension of the low-valence titanium reagent formed was cooled to room temperature and a solution of *o*-nitrobenzamide (3 mmol) and aromatic aldehydes or ketones or cyclic ketone (3 mmol) in THF (10 ml) was added dropwise. The mixture was refluxed 5 h under N_2 (the reaction was monitored by TLC). The reaction mixture was quenched with 10% HCl (50 ml) and extracted with $CHCl_3$ (3×50ml). 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 products **3a–i** and **5a–d** were purified by recrystallisation from 95% ethanol.

2-(4-chlorophenyl)-1, 2-dihydroquinazolin-4(3H)-one (**3a**): M.p. 202–204 ˚C; ^{1}H NMR ($CDCl_3$) δ: 5.86 (s, 1H, NH), 5.90 (s, 1H, C^2-H), 6.68 (d, *J*=8.0 Hz, 1H, ArH), 6.90–6.94 (m, 1H, ArH), 7.33–7.37 (m, 1H, ArH), 7.34 (d, *J*=8.0 Hz, 2H, ArH), 7.55 (d, *J*=8.0 Hz, 2H, ArH), 7.94 (d, *J*=8.0 Hz, 1H, ArH); IR (KBr) *v*: 3309, 3192, 1655, 1609, 1509, 1484, 1434, 1384, 1325, 1292, 1168, 1152, 1134, 1092, 1016, 948, 913, 862, 836, 799, 752, 700 cm^{-1}. Anal. calcd for $C_{14}H_{11}ClN_2O$: C 65.00, H 4.29, N 10.83; found C 65.23, H 4.07, N 10.62.

2-(3,4-dimethoxyphenyl)-1, 2-dihydroquinazolin-4(3H)-one (**3b**): M.p. 214–216 ˚C, ^{1}H NMR ($CDCl_3$) δ: 3.92 (s, 6H, 2×CH_3O), 5.79 (s, 1H, NH), 5.86 (s, 1H, C^2-H), 6.68 (d, *J*=8.0 Hz, 1H, ArH), 6.87–6.93 (m, 2H, ArH), 7.05 (d, *J*=8.0 Hz, 1H, ArH), 7.21 (s, 1H, ArH), 7.33–7.35 (m, 1H, ArH), 7.95 (d, *J*=7.2 Hz, 1H, ArH); IR (KBr) *v*: 3356, 3181, 3076, 2960, 1656, 1613, 1504, 1460, 1423, 1378, 1304, 1261, 1230, 1186, 1140, 1023, 856, 823, 760 cm^{-1}. Anal. calcd for $C_{16}H_{16}N_2O_3$: C 67.59, H 5.67, N 9.85; found C 67.81, H 5.40, N 10.03.

2-(3,4-dimethylenedioxyphenyl)-1, 2-dihydroquinazolin-4(3H)-one (**3c**): M.p. 199–201 ˚C; ^{1}H NMR ($CDCl_3$) δ: 5.80 (s, 1H, NH), 5.82 (s, 1H, C^2-H), 6.02 (s, 2H, OCH_2O), 6.67 (d, *J*=8.4 Hz, 1H, ArH), 6.83 (d, *J*=8.4 Hz, 1H, ArH), 6.88–6.92 (m, 1H, ArH), 6.99 (d, *J*=7.6 Hz, 1H, ArH), 7.15 (s, 1H, ArH), 7.32–7.36 (m, 1H, ArH), 7.94 (d, *J*=7.2 Hz, 1H, ArH); IR (KBr) *v*: 3282, 3181, 1654, 1612, 1486, 1446, 1388, 1327, 1297, 1248, 1187, 1164, 1149, 1121, 1105, 1036, 930, 864, 786, 754 cm^{-1}. Anal. calcd for $C_{15}H_{12}N_2O_3$: C 67.16, H 4.51, N 10.44; found C 67.33, H 4.29, N 10.52.

2-phenyl-1, 2-dihydroquinazolin-4(3H)-one (**3d**): M.p. 215–216 ˚C; ^{1}H NMR ($CDCl_3$) δ: 5.81 (s, 1H, NH), 5.91 (s, 1H, C^2-H), 6.68 (d, *J*=8.4 Hz, 1H, ArH), 6.89–6.93 (m, 1H, ArH), 7.32–7.36 (m, 1H, ArH), 7.45–7.46 (m, 3H, ArH), 7.59–7.61 (m, 2H, ArH), 7.95 (d, *J*=7.6 Hz, 1H, ArH); IR (KBr) *v*: 3303, 3174, 1654, 1613, 1510, 1392, 1300, 1149, 810, 748, 699 cm^{-1}. Anal. calcd for $C_{14}H_{12}N_2O$: C 74.98, H 5.39, N 12.49; found C 75.13, H 5.23, N 12.37.

2-(methylphenyl)-1, 2-dihydroquinazolin-4(3H)-one (**3e**): M.p. 231–233 ˚C; ^{1}H NMR ($CDCl_3$) δ: 2.40 (s, 3H, CH3), 5.78 (s, 1H, NH), 5.87 (s, 1H, C2-H), 6.67 (d, *J*=8.4 Hz, 1H, ArH), 6.88–6.92 (m, 1H, ArH), 7.24–7.26 (m, 2H, ArH), 7.32–7.35 (m, 1H, ArH), 7.48 (d, *J*=7.2 Hz, 2H, ArH), 7.95 (d, *J*=7.2 Hz, 1H, ArH); IR (KBr) *v*: 3312, 3194, 1657, 1611, 1509, 1486, 1438, 1385, 1328, 1297, 1151, 1133, 1022, 948, 909,859, 800, 751 cm^{-1}. Anal. calcd for $C_{15}H_{14}N_2O$: C 75.61, H 5.92, N 11.76; found C 75.83, H 5.78, N 11.90.

7-chloro-2-(4-chlorophenyl)-1, 2-dihydroquinazolin-4(3H)-one (**3f**): M.p. 236–237 ˚C; ^{1}H NMR ($CDCl_3$) δ: 5.83 (s, 1H, NH), 5.90 (s, 1H, C^2-H), 6.68 (s, 1H, ArH), 6.88 (d, *J*=8.8 Hz, 1H, ArH), 7.44 (d, *J*=8.0Hz, 2H, ArH), 7.53 (d, *J*=8.0 Hz, 2H, ArH), 7.86 (d, *J*=8.0 Hz, 1H, ArH); IR (KBr) *v*: 3250, 3181, 1649, 1609, 1471, 1322, 1301, 1248, 1186, 1129, 1090, 1007, 875, 833, 803 cm^{-1}. Anal. calcd for $C_{14}H_{10}Cl_2N_2O$: C 57.36, H 3.44, N 9.56; found C 57.51, H 3.38, N 9.47.

7-chloro-2-(4-fluophenyl)-1, 2-dihydroquinazolin-4(3H)-one (**3g**): M.p. 179–180 ˚C; ^{1}H NMR ($CDCl_3$) δ: 5.88 (s, 1H, NH), 5.90 (s, 1H, C^2-H), 6.68 (s, 1H, ArH), 6.87 (d, *J*=8.0 Hz, 1H, ArH), 7.12–7.16 (m, 2H, ArH), 7.56–7.59 (m, 2H, ArH), 7.85 (d, *J*=8.0 Hz, 1H, ArH); IR (KBr) *v*: 3251, 3181, 1649, 1610, 1508, 1471, 1322, 1301, 1229, 1158, 1129, 1091, 1010, 874, 846, 690 cm^{-1}. Anal. calcd for $C_{14}H_{10}ClFN_2O$: C 60.77, H 3.64, N 10.12; found C 60.91, H 3.57, N 10.03.

7-chloro-2-(4-methylphenyl)-1,2-dihydroquinazolin-4(3H)-one (**3h**): M.p. 242–244 ˚C; ^{1}H NMR ($CDCl_3$) δ: 2.40 (s, 3H, CH3), 5.78 (s, 1H, NH), 5.87 (s, 1H, C^2-H), 6.67 (s, 1H, ArH), 6.86 (d, *J*=8.8 Hz, 1H, ArH), 7.25–7.26 (m, 2H, ArH), 7.46 (d, *J*=8.0 Hz, 2H, ArH), 7.87 (d, *J*=8.8 Hz, 1H, ArH); IR (KBr) *v*: 3294, 3181, 1654, 1606, 1510, 1471, 1432, 1370, 1297, 1172, 1133, 1079, 1014, 922, 858, 816, 748, 721, 678 cm^{-1}. Anal. calcd for $C_{15}H_{13}ClN_2O$: C 66.06, H 4.80, N 10.27; found C 66.31, H 4.63, N 10.47.

7-chloro-2-(4-methoxyphenyl)-1,2-dihydroquinazolin-4(3H)-one (**3i**): M.p. 215–216 ˚C; ^{1}H NMR ($CDCl_3$) δ: 3.85 (s, 3H, OCH_3), 5.77 (s, 1H, NH), 5.85 (s, 1H, C^2-H), 6.67 (s, 1H, ArH), 6.86 (d, *J*=8.0 Hz, 1H, ArH), 6.96 (d, *J*=8.8 Hz, 2H, ArH), 7.50 (d, *J*=8.8 Hz, 2H, ArH), 7.86 (d, *J*=8.0 Hz, 1H, ArH); IR (KBr) *v*: 3295, 3185, 1654, 1610, 1510, 1480, 1427, 1369, 1294, 1254, 1171, 1135, 1110, 1079, 1038, 923, 863, 834, 782, 748, 680 cm^{-1}. Anal. calcd for $C_{15}H_{13}ClN_2O_2$: C 62.40, H 4.54, N 9.70; found C 62.63, H 4.38, N 9.63.

2,2-dimethyl-1, 2-dihydroquinazolin-4(3H)-one (**3j**): M.p. 185–186 ˚C; ^{1}H NMR ($CDCl_3$) δ: 1.57 (s, 6H, 2×CH_3), 6.30 (s, 1H, NH), 6.63 (d, *J*=8.0 Hz, 1H, ArH), 6.82–6.85 (m, 1H, ArH), 7.29–7.33 (m, 1H, ArH), 7.89 (d, *J*=8.0 Hz, 1H, ArH); IR (KBr) *v*: 3260, 3180, 2968, 1634, 1608, 1520, 1486, 1424, 1392, 1362, 1334, 1279, 1182, 753 cm^{-1}. Anal. calcd for $C_{10}H_{12}N_2O$: C 68.16, H 6.86, N 15.90; found C 68.30, H 6.63, N 15.73.

2-methyl-2-ethyl-1, 2-dihydroquinazolin-4(3H)-one (**3k**): M.p. 184–185 ˚C; ^{1}H NMR ($CDCl_3$) δ: 0.99 (t, *J*=8.0 Hz, 3H, CH_3), 1.50 (s, 3H, CH_3), 1.81 (q, *J*=8.0 Hz, 2H, CH_2), 6.16(s, 1H, NH), 6.62 (d, *J*=8.0 Hz, 1H, ArH), 6.79–6.83 (m, 1H, ArH), 7.27–7.32 (m, 1H, ArH), 7.87 (d, *J*=8.0 Hz, 1H, ArH); IR (KBr) *v*: 3279, 3178, 2974, 1643, 1609, 1512, 1489, 1430, 1395, 1331, 1275, 1182, 1153, 758 cm^{-1}. Anal. calcd for $C_{11}H_{14}N_2O$: C 69.45, H 7.42, N 14.73; found C 69.61, H 7.38, N 14.54.

2,2-diethyl-1,2-dihydroquinazolin-4(3H)-one (**3l**): M.p. 190–191 ˚C; ^{1}H NMR ($CDCl_3$) δ: 0.97 (t, *J*=8.0 Hz, 6H, 2×CH_3), 1,76 (q, *J*=8.0 Hz, 4H, 2×CH_2), 5.98 (s, 1H, NH), 6.60 (d, *J*=8.0 Hz, 1H, ArH), 6.76–6.80 (m, 1H, ArH), 7.27–7.30 (m, 1H, ArH), 7.85 (d, *J*=8.0 Hz,1H, ArH); IR (KBr) *v*: 3320, 3175, 2974, 1646, 1607, 1510, 1489, 1463, 1429, 1395, 1329, 1274, 1150, 758 cm^{-1}. Anal. calcd for $C_{12}H_{16}N_2O$: C 70.56, H 7.90, N 13.71; found C 70.81, H 7.68, N 13.59.

7-chloro-2,2-dimethyl-1,2-dihydroquinazolin-4(3H)-one (**3m**): M.p. 243–244 ˚C; ^{1}H NMR ($CDCl_3$) δ: 1.58 (s, 6H, 2×CH_3), 6.21 (s, 1H, NH), 6.64 (s, 1H, ArH), 6.80 (d, *J*=8.8 Hz, 1H, ArH), 7.81 (d, *J*=8.8 Hz, 1H, ArH); IR (KBr) *v*: 3291, 3178, 2975, 1643, 1607, 1510, 1480, 1458, 1416, 1387, 1363, 1276, 1244, 1178, 1078, 984, 857, 777 cm^{-1}. Anal. calcd for $C_{10}H_{11}ClN_2O$: C 57.01, H 5.26, N 13.30; found C 57.27, H 5.14, N 13.18.

7-chloro-2-methyl-2-ethyl-1,2-dihydroquinazolin-4(3H)-one (**3n**): M.p. 173–174 ˚C; ^{1}H NMR ($CDCl_3$) δ: 0.98 (t, *J*=8.0 Hz, 3H, CH_3), 1.50 (s, 3H, CH_3), 1.79 (q, *J*=8.0 Hz, 2H, CH_2), 6.29 (s, 1H, ArH), 6.75 (d, *J*=8.0 Hz, 1H, ArH), 6.96 (s, 1H, NH), 7.78 (d, *J*=8.0 Hz, 1H, ArH); IR (KBr) *v*: 3304, 3191, 2974, 1642, 1608, 1510, 1480, 1454, 1419, 1320, 1276, 1156, 1079, 896, 854, 778 cm^{-1}. Anal. calcd for $C_{11}H_{13}ClN_2O$: C 58.80, H 5.83, N 12.47; found C 58.94, H 5.62, N 12.33.

7-chloro-2,2-diethyl-1,2-dihydroquinazolin-4(3H)-one (**3o**): M.p. 164–166˚ C; ^{1}H NMR ($CDCl_3$) δ: 0.96 (t, *J*=8.0 Hz, 6H, 2×CH_3), 1.75 (q, *J*=8.0 Hz, 4H, 2×CH_2), 6.59 (s, 1H, NH), 6.61 (s, 1H, ArH), 6.71 (d, *J*=8.0 Hz, 1H, ArH), 7.76 (d, *J*=8.0 Hz, 1H, ArH); IR (KBr) *v*: 3286, 3215, 2967, 1644, 1608, 1513, 1483, 1461, 1420, 1363, 1324, 1276, 1175, 1155, 1082, 986, 912, 874, 774 cm^{-1}. Anal. calcd for $C_{12}H_{15}ClN_2O$: C 60.38, H 6.33, N 11.74; found C 60.56, H 6.18, N 11.59.

2,2-tetramethylene-1,2-dihydroquinazolin-4(3H)-one (**5a**): M.p. 251–253 ˚C; ^{1}H NMR ($CDCl_3$) δ: 1.79–1.80 (m, 4H, CH_2CH_2), 1.90–1.97 (m, 4H, 2×CH_2), 6.17 (s, 1H, NH), 6.65 (d, *J*=8.0 Hz, 1H, ArH), 6.85 (t, *J*=7.2 Hz, 1H, ArH), 7.31 (t, *J*=7.2 Hz, 1H, ArH), 7.88 (d, *J*=8.0 Hz, 1H, ArH); IR (KBr) *v*: 3292, 3159, 2972, 1638, 1606, 1517, 1485, 1431, 1385, 1334, 1270, 1149, 1088, 1049, 954, 849, 803, 781, 753 cm^{-1}. Anal. calcd for $C_{12}H_{14}N_2O$: C 71.26, H 6.98, N 13.85; found C 71.48, H 6.78, N 13.72.

7-chloro-2,2-tetramethylene-1,2-dihydroquinazolin-4(3H)-one (**5b**): M.p. 244–245 ˚C; ^{1}H NMR ($CDCl_3$) δ: 1.62–1.80 (m, 4H, 2×CH_2), 1.81–1.96 (m, 4H, 2×CH_2), 6.23 (s, 1H, NH), 6.65 (s, 1H, ArH), 6.80 (d, *J*=8.8 Hz, 1H, ArH), 7.80 (d, *J*=8.8 Hz, 1H, ArH); IR (KBr) *v*: 3260, 3189, 1650, 1608, 1519, 1480, 1421, 1361, 1318, 1277, 1154, 1078, 1044, 936, 899, 855, 768 cm^{-1}. Anal. calcd for $C_{12}H_{13}ClN_2O$: C 60.89, H 5.54, N11.84; found C 61.03, H 5.36, N 11.63.

2,2-pentamethylene-1,2-dihydroquinazolin-4(3H)-one (**5c**): M.p. 224–225 ˚C; ^{1}H NMR ($CDCl_3$) δ: 1.47–1.48 (m, 2H, CH_2), 1.53–1.60 (m, 4H, 2×CH_2), 1.80–1.84 (m, 4H, 2×CH_2), 6.19 (s, 1H, NH), 6.65 (d, *J*=8.0 Hz, 1H, ArH), 6.82 (t, *J*=7.2 Hz, 1H, ArH), 7.30 (t, *J*=7.2 Hz, 1H, ArH), 7.87 (d, *J*=8.0 Hz, 1H, ArH); IR (KBr) *v*: 3367, 3170, 2923, 1651, 1612, 1507, 1484, 1417, 1382, 1323, 1269, 1210, 1178, 1145, 1093, 1040, 1004, 951, 914, 855, 802, 760 cm^{-1}. Anal. calcd for $C_{13}H_{16}N_2O$: C 72.19, H 7.46, N 12.95; found C 72.37, H 7.23, N 13.09.

7-chloro-2,2-pentamethylene-1,2-dihydroquinazolin-4(3H)-one (**5d**): M.p. 221–222 ˚C; ^{1}H NMR ($CDCl_3$) δ: 1.43–1.52 (m, 2H, CH_2), 1.52–1.68 (m, 4H, 2×CH_2), 1.74–1.90 (m, 4H, 2×CH_2), 6.17 (s, 1H, NH), 6.66 (s, 1H, ArH), 6.77 (d, *J*=8.8 Hz, 1H, ArH), 7.78 (d, *J*=8.8 Hz, 1H, ArH); IR (KBr) *v*: 3362, 3249, 1699, 1600, 1576, 1507, 1464, 1336, 1152, 1044, 890, 751 cm^{-1}. Anal. calcd for $C_{13}H_{15}ClN_2O$: C 62.28, H 6.03, N 11.17; found C 62.51, H 5.92, N 11.34.

Crystal data and structure refinement for 5c: Crystals suitable for X-ray analysis were obtained by slow evaporation of an ethanol solution of 5c. $C_{13}H_{16}N_2O$, *M* = 216.28, monoclinic, space group *P*2(1)/n, *a* = 10.387(1), *b* = 10.954(2), *c* = 10.827(2) Å, β = 110.77(1)°, *V* = 1151.8(4) Å^3, *Z* = 4, *D*c = 1.247 gcm^{-3}, *F*(000) = 464, μ (Mo*K*α) = 0.080 mm^{-1}, colourless block crystals, crystal size 0.52×0.48×0.44 mm.

Intensity data were collected at 296 K on a Siemens P4 diffractometer with graphite-monochromated Mo*K*α radiation (λ = 0.71073 Å): 2033 independent reflections were collected using ω scan mode in the range of 2.34° <θ <25.00°, of which 1575 intensity data with [*I*>2σ(*I*)] were observed. The corrections for *Lp* factors were applied. The structure was solved by direct methods and expanded using Fourier techniques. Some non-hydrogen atoms were refined anisotropically, while the rest were refined isotropically. Hydrogen atoms were induded in F value calculation but fixed during the structure refinement. A full-matrix least-squares refinement gave final *R* = 0.0449 and *wR* = 0.1217 with $w = 1/[\sigma^2 Fo^2 + (0.0631P)^2 + 0.2555P]$, *S* = 1.087. The maximum peak in the difference Fourier map is 0.296 eÅ^{-3} and the minimum peak is –0.310 eÅ^{-3}. In the final circle refinement the largest parameter shifts $(\Delta/\sigma)_{max}$ is 0.000. Crystallographic data (excluding structure factors) for the structure reported in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication No. CCDC-201813.

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