A highly efficient method for the synthesis of novel 1'*H*-spiro[indene-2,2'-quinazoline]-1,3,4'(3'*H*)-trione derivatives

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A series of novel ninhydrin-derived spiro-quinazolinone derivatives in moderate to good yields have been synthesised through a ferric chloride catalysed reaction in 1,2-dichloroethane.

Keywords: spiro compounds, primary amines, ninhydrin, ferric chloride

Nitrogenous heterocycles are found in the core structure of numerous natural products and pharmaceutical agents. Therefore, much effort has been devoted to access new nitogen-containing cycles. The resultant compounds could be utilised as potential bioactive entities in drug discovery. The interesting biologically active molecules, quinazoline and quinazolinones exhibit diverse properties.[1,2] For instance, gefitinib[3] **1** and raltitrexed[4] **2** have been described as EGFR (epidermal growth factor receptor) inhibitors and antitumour agents respectively. Moreover, spiro-based heterocycles are of high interest to medicinal chemists for their prevalence in many bioactive molecules. The asymmetric spiro carbon atom often gives the molecule special stereochemical features required for interactions with biological systems. As such, spiro quinazolinone systems have versatile pharmacological properties such as the potential inhibitory activity against SIRT1 of **3** [5] and the antitumour character of **4**.[6]

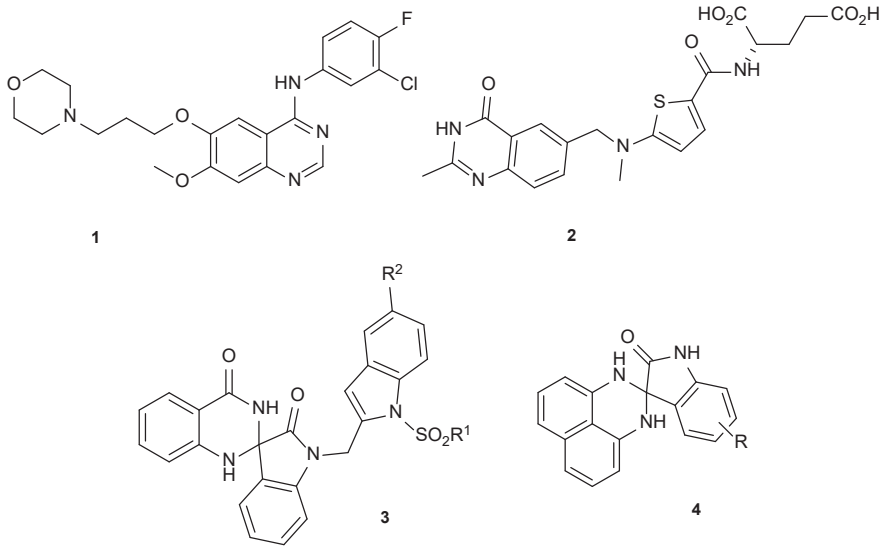
The most well-established strategy for the synthesis of 2,3-dihydro quinazolinones has been based on the condensation of 2-aminobenzamides with carbonyl functionality employing different catalysts like $CuCl_2$,[7] $TiCl_4$-Zn,[8] *p*-TsOH,[9] NH_4Cl,[10] β-cyclodextrin-SO_3H,[11] TCT,[12] I_2 [13] and acidic SiO_2.[14] Furthermore, utilising isatoic anhydride[15-19] and 2-nitrobenzamides[20] with the assistance of the appropriate reducing agent, provides a route for an alternative method of construction of quinazolines.

Focused on 2-aminobenzamide chemistry[21] and concerned with the synthesis of new heterocyclic compounds,[22-27] we now report the $FeCl_3$-catalysed synthesis of 1'*H*-spiro[indene-2,2'-quinazoline]-1,3,4'(3'*H*)-trione derivatives from isatoic anhydride **5** and amines **6**, and the reaction of various 2-amino-*N*-substituted benzamides **7a–h** and ninhydrin **8** in 1,2-dichloroethane.

Results and discussion

As mentioned above, the activity of iodine in similar reactions encouraged us to examine other Lewis acids. So, we carried out the model reaction by taking **8** (1 equiv.) and 2-amino-*N*-benzyl benzamide **7a** (1 equiv.). Benzamide derivatives were prepared by the simple reaction between isatoic anhydride **5** and amine derivatives **6a–h** in water at room temperature (Scheme 1).[28,29]

Screening of the model reaction was conducted using different solvents, Lewis acids and varying temperature as indicated in Table 1. The transformation catalysed by $FeCl_3$ in refluxing 1,2-dichloroethane (DCE) afforded the expected product in 64% yield, whereas by applying CuI, CuBr and $CuBr_2$ as the catalyst, the yields dropped to 59%, 36%, 27% respectively (entries 2–4). In contrast to other solvents such as CH_3CN, DMF, and 1,4-dioxane (entries 5–7), DCE was found to be the solvent of choice based on isolated yields. There was no noticeable improvement in the reaction yield when the reaction was attempted at lower temperatures (entry 8). Next, varied



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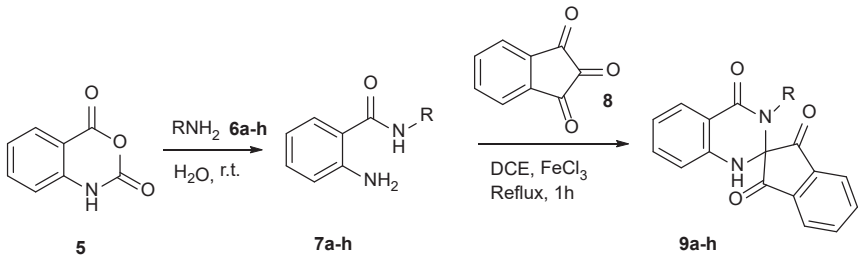
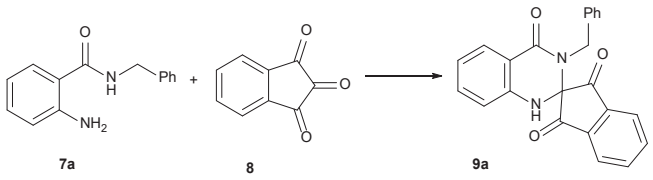


Scheme 1

Table 1 Effects of various conditions on the model reaction



Entry	Solvent	Lewis acid /mol%	Temp./°C	Yield/%[b]
1	DCE	$FeCl_3$ (20)	Reflux	64
2	DCE	CuI (20)	Reflux	59
3	DCE	CuBr (20)	Reflux	36
4	DCE	$CuBr_2$ (20)	Reflux	27
5	CH_3CN	$FeCl_3$ (20)	Reflux	38
6	DMF	$FeCl_3$ (20)	80	29
7	1,4-Dioxane	$FeCl_3$ (20)	Reflux	47
8[c]	DCE	$FeCl_3$ (20)	25	30
9	DCE	$FeCl_3$ (10)	Reflux	51
10	DCE	$FeCl_3$ (30)	Reflux	57
11[c]	DCE	–	Reflux	Trace

[a]All reactions were run with ninhydrin (1 mmol), 2-amino-*N*-benzylbenzamide (1 mmol) and Lewis acid (x mol%) in different solvents (5 mL) for 1h.
[b]Isolated yield.
[c]Reaction time 12 h.

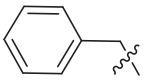
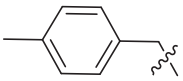
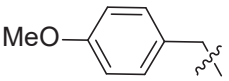
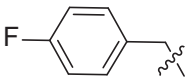
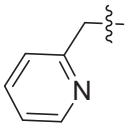
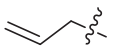
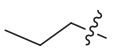
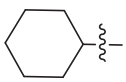
amounts of anhydrous $FeCl_3$ were examined under optimised conditions which revealed 20 mol% as the optimum amount to promote the formation of the spiro-based product (entries 9 and 10). According to our observations complete reaction required the presence of 20 mol% $FeCl_3$ (entry 11).

Proceeding to explore the scope of the reaction after finding the optimised reaction conditions, **8** was treated with the derivatives **7a–h** to synthesise the target compounds. The aliphatic, electron-rich and electron-deficient aromatic amines could be efficiently employed affording the respective spiro-fused ninhydrin-quinazolinones **9a–h** in moderate to good yields (Table 2). The presence of electron-donating substituents seems beneficial for the transformation giving the corresponding products in higher yields. Structures of compounds **9a–h** were characterised by IR, ^{1}H, ^{13}C NMR spectra, MS, and analytical data. As a representative case, the mass spectrum of **9b** exhibited a molecular ion peak at *m/z* = 382 along with a parent ion peak at *m/z* = 277 resulting from C–N bond cleavage. The presence of the NH signal at δ (H) 6.65 ppm in ^{1}H NMR and three signals at 70.7, 162.8 and 194.5 ppm arising from the spiro carbon and carbonyl groups in the ^{13}C NMR spectrum, confirmed the proposed structure.

Conclusion

In summary, we have developed an efficient reaction of ninhydrin with 2-amino-*N*-substituted benzamides bearing electron-donating and electron-withdrawing substituents. The easy synthetic access to the interesting spiro quinazolinone core through commercially accessible starting materials highlights the potential value of these compounds in the construction of biologically active molecules.

Table 2 The reaction scope for the synthesis of 1'*H*-spiro[indene-2,2'-quinazoline]-1,3,4'(3'*H*)-trione (Scheme 1)

Entry	R	Product	Yield/%
1		**9a**	64
2		9b	73
3		**9c**	79
4		**9d**	58
5		**9e**	68
6		**9f**	70
7		**9g**	66
8		**9h**	73

[a]The reactions were run on 1 mmol scale in 1,2-dichloroethane (5 mL) at reflux temperature for 1 h.
[b]Isolated yield.

Experimental

Melting points were taken on a Kofler hot stage apparatus and are uncorrected. ^{1}H and ^{13}C NMR spectrum was recorded on Bruker FT-500, using TMS as an internal standard. The elemental analysis was performed with an Elementar Analysen system GmbH VarioEL CHNS mode. Mass spectra were determined on an Agilent Technology (HP) mass spectrometer operating at an ionisation potential of 70 eV. All reagents and solvents were purchased from Aldrich and Merck, and used without any purification.

*Synthesis of 1'*H*-spiro[indene-2,2'-quinazoline]-1,3,4'(3'*H*)-triones (***9a–h***); general procedure*

$FeCl_3$ (20 mol%) was added to the stirring mixture of ninhydrin (1 mmol) and 2-amino-*N*-substituted benzamides (1 mmol) in 1,2-dichloroethane (5 mL) and refluxed for 1 h. On completion, the solvent was removed and CH_2Cl_2 (15 mL) was added to the residue, washed with water (10 mL), brine (5 mL) and dried over anhydrous

Na_2SO_4. The solvent was removed under reduced pressure and the crude mass was subjected to column chromatography eluting with petroleum ether/ethyl acetate (10:1) to furnish **9a–h** as orange solids.

*3'-Benzyl-1'*H-*spiro[indene-2,2'-quinazoline]-1,3,4'(3'*H*)-trione* (**9a**): Yield 64%; m.p. 170–172 °C; IR (KBr): 3345, 1720, 1679, 1381, 1294, 1194 cm^{-1}; ^{1}H NMR (500 MHz, DMSO-d_6): δ 8.00 (dd, *J* = 5.5, 3.0 Hz, 2H), 7.78–7.89 (m, 2H), 7.77 (d, *J* = 7.8 Hz, 1H), 7.72 (s, 2H), 7.29 (t, *J* = 7.8 Hz, 1H), 7.14 (d, *J* = 8.4 Hz, 1H), 6.79–6.83 (m, 2H), 6.57 (d, *J* = 8.0 Hz, NH), 6.49 (d, *J* = 8.4 Hz, 2H), 4.55 (s, NCH_2) ppm; ^{13}C NMR (125 MHz, DMSO-d_6): δ 48.1 (NCH_2), 72.0 (C_{spiro}), 123.7, 123.9, 124.0, 124.1, 124.6, 131.2, 137.0, 137.3, 137.5, 137.8, 138.5, 138.8, 139.3, 161.9, 193.6 ppm; MS (70 eV): *m/z* = 368 (M^+, 74), 339 (29), 277 (100), 255 (81), 91 (56), 76 (43); Anal calcd for $C_{23}H_{16}N_2O_3$: C, 74.99; H, 4.38; N, 7.60; found: C, 74.77; H, 4.49; N, 7.49%.

*3'-(4-Methylbenzyl)-1'*H-*spiro[indene-2,2'-quinazoline]-1,3,4'(3'*H*)-trione* (**9b**): Yield 73%; m.p. 159–161 °C; IR (KBr): 3310, 1729, 1662, 1361, 1241, 841 cm^{-1}; ^{1}H NMR (500 MHz, DMSO-d_6): δ 7.99 (d, *J* = 7.2 Hz, 1H), 7.72 (d, *J* = 8.0 Hz, 1H), 7.62 (t, *J* = 7.5 Hz, 1H), 7.50 (t, *J* = 7.2 Hz, 1H), 7.38 (t, *J* = 7.8 Hz, 1H), 7.32 (d, *J* = 7.5 Hz, 1H), 7.12 (t, *J* = 7.4 Hz, 1H), 6.92 (d, *J* = 7.8 Hz, 3H), 6.65 (s, NH), 6.60 (d, *J* = 7.8 Hz, 2H), 4.70 (s, NCH_2), 2.22 (s, CH_3) ppm; ^{13}C NMR (125 MHz, DMSO-d_6): δ 20.7 (CH_3), 43.8 (NCH_2), 70.7 (C_{spiro}), 113.3, 114.4, 123.2, 125.9, 126.0, 127.0, 127.4, 128.5, 129.1, 130.7, 131.0, 135.5, 142.6, 162.8, 194.5 ppm; MS (70 eV): *m/z* = 382 (M^+, 43), 366 (21), 277 (100), 105 (71), 76 (67), 45 (32). Anal calcd for $C_{24}H_{18}N_2O_3$: C, 75.38; H, 4.74; N, 7.33; found: C, 75.19; H, 4.63; N, 7.47%.

*3'-(4-Methoxybenzyl)-1'*H-*spiro[indene-2,2'-quinazoline]-1,3,4'(3'*H*)-trione* (**9c**): Yield 79%; m.p. 120–122 °C; IR (KBr): 3360, 1738, 1645, 1350, 1291, 809 cm^{-1}; ^{1}H NMR (500 MHz, DMSO-d_6): δ 8.40 (s, 1H), 8.16 (m, 1H), 8.00–8.08 (m, 5H), 7.86–7.90 (m, 1H), 7.83 (d, *J* = 7.7 Hz, 1H), 7.78 (d, *J* = 7.7 Hz, 1H), 7.72 (t, *J* = 7.4 Hz, 1H), 7.55 (s, 1H), 6.68 (s, NH), 4.57 (s, NCH_2), 3.44 (s, OMe) ppm; ^{13}C NMR (125 MHz, DMSO-d_6): δ 44.1 (NCH_2), 56.0 (OCH_3), 72.0 (C_{spiro}), 122.7, 123.5, 124.0, 124.6, 131.2, 132.5, 136.4, 137.0, 138.5, 139.3, 140.7, 148.7, 158.7, 163.5, 193.6 ppm; MS (70 eV): *m/z* = 398 (M^+, 34), 367 (57), 277 (100), 121 (68), 76 (47), 32 (29). Anal calcd for $C_{24}H_{18}N_2O_4$: C, 72.35; H, 4.55; N, 7.03; found: C, 72.19; H, 4.69; N, 6.89%.

*3'-(4-Fluorobenzyl)-1'*H-*spiro[indene-2,2'-quinazoline]-1,3,4'(3'*H*)-trione* (**9d**): Yield 58%; m.p. 200–202 °C; IR (KBr): 3376, 1726, 1675, 1429, 1316, 1291, 845 cm^{-1}; ^{1}H NMR (500 MHz, DMSO-d_6): δ 8.05 (dd, *J* = 5.7, 3.1 Hz, 2H), 7.94 (dd, *J* = 5.7, 3.1 Hz, 2H), 7.75–7.77 (m, 2H), 7.30 (t, *J* = 6.9 Hz, 1H), 6.98–7.00 (m, 2H), 6.80–6.87 (m, 3H), 6.58 (d, *J* = 7.9 Hz, NH), 4.51 (s, NCH_2) ppm; ^{13}C NMR (125 MHz, DMSO-d_6): δ 45.0 (NCH_2), 73.6 (C_{spiro}), 114.0, 114.5 (d, $J_{C\text{-}F}$ = 21 Hz), 118.6, 124.1, 127.7, 128.7, 130.4 (d, $J_{C\text{-}F}$ = 8 Hz), 132.2, 133.9, 137.8, 138.9, 145.2, 160.3, 162.3 (d, $J_{C\text{-}F}$ = 129 Hz), 194.3 ppm; MS (70 eV): *m/z* = 386 (M^+, 52), 357 (17), 277 (100), 250 (35), 109 (86), 76 (26), 50 (9). Anal calcd for $C_{23}H_{15}FN_2O_3$: C, 71.50; H, 3.91; N, 7.25; found: C, 71.39; H, 3.79; N, 6.99%.

*3'-(Pyridin-2-ylmethyl)-1'*H-*spiro[indene-2,2'-quinazoline]-1,3,4'(3'*H*)-trione* (**9e**): Yield 68%; m.p. 212–214 °C; IR (KBr): 3300, 1730, 1665, 1381, 1249 cm^{-1}; ^{1}H NMR (500 MHz, DMSO-d_6): δ 8.31 (m, 1H), 8.18 (s, 1H), 8.08–8.09 (m, 2H), 8.00 (m, 2H), 7.85 (s, 1H), 7.76 (d, *J* = 7.5 Hz, 1H), 7.50 (d, *J* = 7.6 Hz, 1H), 7.31 (t, *J* = 7.5 Hz, 1H), 7.17 (s, 1H), 6.82 (t, *J* = 7.4 Hz, 1H), 6.60 (d, *J* = 7.9 Hz, NH), 4.49 (s, NCH_2) ppm; ^{13}C NMR (125 MHz, DMSO-d_6): δ 44.0 (NCH_2), 74.2 (C_{spiro}), 113.5, 114.1, 118.7, 123.1, 124.4, 127.6, 132.3, 134.1, 135.6, 138.0, 139.0, 145.2, 148.3, 149.1, 163.6, 194.3 ppm; MS (70 eV): *m/z* = 369 (M^+, 54), 340 (25), 277 (100), 92 (64), 76 (83). Anal calcd for $C_{22}H_{15}N_3O_3$: C, 71.54; H, 4.09; N, 11.38; found: C, 71.40; H, 4.16; N, 11.58%.

*3'-Allyl-1'*H-*spiro[indene-2,2'-quinazoline]-1,3,4'(3'*H*)-trione* (**9f**): Yield 70%; m.p. 188–190 °C; IR (KBr): 3312, 1720, 1679, 1394, 1276 cm^{-1}; ^{1}H NMR (500 MHz, DMSO-d_6): δ 8.09–8.14 (m, 4H), 7.80 (s, 1H), 7.71 (d, *J* = 7.8 Hz, 1H), 7.29 (t, *J* = 7.6 Hz, 1H), 6.80 (t, *J* = 7.5 Hz, 1H), 6.57 (d, *J* = 8.0 Hz, NH), 5.47–5.53 (m, 1H), 4.77 (d, *J* = 10.2 Hz, 1H), 4.68–4.72 (m, 1H), 3.92 (d, *J* = 6.4 Hz, NCH_2) ppm; ^{13}C NMR (125 MHz, DMSO-d_6): δ 45.0 (NCH_2), 73.8 (C_{spiro}), 113.9, 114.2, 118.5, 119.1, 124.3, 127.5, 133.0, 133.8, 138.0, 139.1, 145.0, 162.7, 194.5 ppm; MS (70 eV): *m/z* = 318 (M^+, 31), 301 (25), 277 (100), 261 (52), 185 (77), 105 (62), 76 (86), 41 (64). Anal calcd for $C_{19}H_{14}N_2O_3$: C, 71.69; H, 4.43; N, 8.80; found: C, 71.80; H, 4.33; N, 8.85%.

*3'-Propyl-1'*H-*spiro[indene-2,2'-quinazoline]-1,3,4'(3'*H*)-trione* (**9g**): Yield 66 %; m.p. 123–125 °C; IR (KBr): 3381, 1755, 1649, 1378, 1254 cm^{-1}; ^{1}H NMR (500 MHz, DMSO-d_6): δ 7.93 (d, *J* = 8.0 Hz, 1H), 7.69 (d, *J* = 8.1 Hz, 1H), 7.60 (t, *J* = 7.7 Hz, 1H), 7.51–7.54 (m, 2H), 7.46 (t, *J* = 7.2 Hz, 1H), 7.24 (t, *J* = 7.7 Hz, 2H), 6.54 (s, NH), 2.77–2.83 (m, 2H), 1.05–1.12 (m, 2H), 0.56 (t, *J* = 7.3 Hz, 3H) ppm; ^{13}C NMR (125 MHz, DMSO-d_6): δ 10.8 (CH_2), 21.8 (CH_3), 42.2 (NCH_2), 68.7 (C_{spiro}), 114.5, 123.6, 127.0, 127.2, 127.3, 129.4, 130.6, 131.2, 142.4, 162.7, 193.8 ppm; MS (70 eV): *m/z* = 320 (M^+, 69), 303 (19), 277 (100), 156 (41), 76 (34), 43(76). Anal calcd for $C_{19}H_{16}N_2O_3$: C, 71.24; H, 5.03; N, 8.74; found: C, 71.30; H, 5.13; N, 8.56%.

*3'-Cyclohexyl-1'*H-*spiro[indene-2,2'-quinazoline]-1,3,4'(3'*H*)-trione* (**9h**): Yield 73%; m.p. 110–112 °C; IR (KBr): 3296, 1771, 1645, 1376, 1225 cm^{-1}; ^{1}H NMR (500 MHz, DMSO-d_6): δ 7.88 (dd, *J* = 7.9, 1.2 Hz, 1H), 7.61 (dt, *J* = 6.9, 1.4 Hz, 1H), 7.51 (t, *J* = 7.3 Hz, 1H), 7.29–7.31 (m, 2H), 7.24 (t, *J* = 7.3 Hz, 1H), 7.12 (t, *J* = 7.3 Hz, 1H), 6.74 (d, *J* = 7.0 Hz, 1H), 6.67 (s, NH), 4.05–4.07 (m, 1H), 1.83–1.85 (m, 2H), 1.67–1.75 (m, 2H), 1.36–1.42 (m, 1H), 1.21–1.34 (m, 4H), 1.06–1.11 (m, 1H) ppm; ^{13}C NMR (125 MHz, DMSO-d_6): δ 24.7, 25.3, 31.9, 50.0 (NCH), 68.8 (C_{spiro}), 116.0, 121.6, 124.5, 126.5, 128.4, 128.6, 134.3, 135.5, 148.9, 161.9, 194.0 ppm; MS (70 eV): *m/z* = 360 (M^+, 55), 343 (41), 277 (100), 167 (49), 83 (31), 76 (38). Anal calcd for $C_{22}H_{20}N_2O_3$: C, 73.32; H, 5.59; N, 7.77; found: C, 73.18; H, 5.63; N, 7.64%.

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