Superacid catalysed the one-pot synthesis of spiro[indole-3,9'-tetrazolo[5,1-*b*]quinazoline]-2,8'(1*H*, 5'*H*)-dione in aqueous medium

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An efficient, one-pot synthesis of spiro[indole-3,9'-tetrazolo[5,1-*b*]quinazoline]-2,8'(1*H*,5'*H*)-dione derivatives by the reaction of isatin, 5-amino-1H-tetrazole, and 5,5-dimethylcyclohexane-1,3-dione catalysed by a superacid ($[MeC(OH)_2]+ClO_4^-$) in aqueous medium is described. This protocol provides a simple one-step procedure with the advantages of easy work-up, high yield, mild reaction conditions and is environmental friendly.

Keywords: spirooxindole, 5-amino-1*H*-tetrazole, multicomponent reactions, superacid

The heterocyclic spirooxindole skeleton is an important structure in organic and medicinal chemistry due to its potency and variety of applications such as antifungal,[1] anti-mycobacterial,[2] anti-tumour,[3] anti-oxidant,[4] antimicrobial,[5] and other biological activities[6] and can also serve as synthons for naturally occurring alkaloids and pharmaceutically important drug molecules.[7]

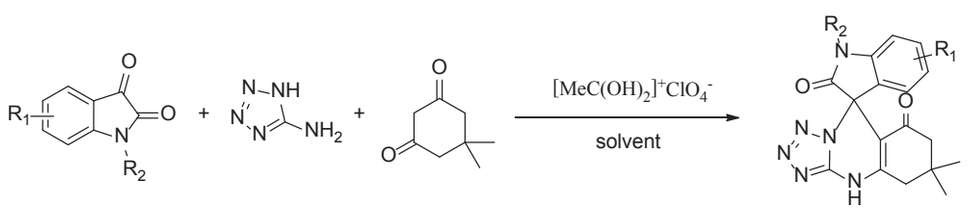
Multicomponent reactions (MCRs) have attracted much attention for the construction of bioactive heterocyclic compounds by virtue of their convergence, productivity, facile execution, and generally high yields of products.[8] A series of efficient methods have been reported for the synthesis of compounds bearing heterocyclic spirooxindole skeletons *via* multicomponent condensation reactions in good to excellent yields.[9–13] A recent development in this field is the incorporation of different functional groups or moieties such as 5-aminopyrazoles,[9] trifluoromethyl,[10] and thiazolidinone[13] into the heterocyclic spirooxindole ring system to achieve good bioactivity. On the other hand, the tetrazole ring system also exhibits a wide range of bioactivities,[14,15] and has been used as an activator[16] and in anticonvulsants[17]. Thus, heterocyclic systems resulting from the annulations of the tetrazole ring on a biologically versatile oxindole moiety in a single molecular framework may increase their biological activities or produce new medicinal properties.

As part of our on-going research programs directed at the development of efficient methodologies for the preparation of heterocyclic compounds and our interest in the environmentally friendly synthetic methodologies, we now report a fast and efficient protocol for the synthesis of spiroheterocycles containing both tetrazole ring and oxindole ring in the presence of catalytic amount of superacid ($[MeC(OH)_2]^+ClO_4^-$), which is simply prepared by mixing acetic acid and perchloric acid at room temperature.[18,19] (Scheme 1) To the best of our knowledge, this is the first report of the synthesis of spiroheterocycles containing both tetrazole ring and oxindole rings.

Initially, the reaction of isatin, 5-amino-1*H*-tetrazole and dimedone was carried out to optimise the reaction conditions (Table 1). First, several solvents such as DCM, toluene, MeOH, EtOH and water were tested. Table 1 reveals that the solvent played an important role. Generally, polar solvents were more effective than the non-polar ones. However, when the reaction proceeded in pure water, only moderate yield (65%, entry 7) was obtained, possibly due to the lesser homogeneity of the reaction mixture. Results clearly showed that $EtOH/H_2O$ (1:1, v/v) was the best choice, affording the corresponding product **4a** in 90% yield within 3 hours (entry 8). Similarly, the acid catalysts also showed a large influence on the reaction. It was observed that the model reaction was very sluggish in the absence of any catalyst. Some strong Brønsted acids including conc. H_2SO_4, $HClO_4$, and toluene-*p*-sulfonic acid could promote the reaction, however, the reaction time was long and the yields were moderate. The most optimal quantity of catalyst seemed to be 10 mol% of $[MeC(OH)_2]^+ClO_4^-$.

With the optimised reaction conditions obtained, the generality and functional group tolerance of this protocol were investigated for this MCR. As shown in Table 2, it was obvious that this method showed good substrate compatibility for isatin. Different electron-withdrawing groups as well as methyl in the 5-, 6-, and 7- positions showed negligible influences, providing the products in good to excellent yields. Moreover, we found that different N-substituted isatins were also compatible with this protocol, giving product **4i** and **4j** in 89% and 86% yield, respectively.

The plausible reaction mechanism was outlined in Scheme 2. In the first step, condensation of isatin and dimedone was proposed to give intermediate **A**, which was further attacked by 5-amino-1*H*-tetrazole to provide intermediate **B**. Finally, the intermediate **B** underwent intramolecular cyclisation to form the target product **4a**.



Scheme 1 Superacid catalysed the one-pot synthesis of spiroheterocycles.

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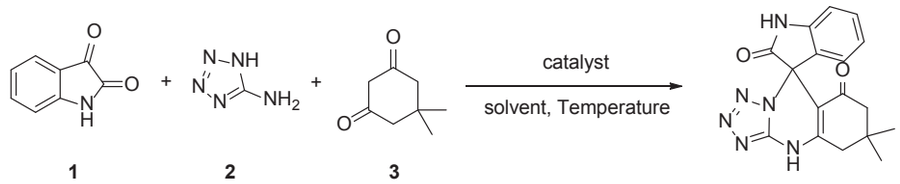
Table 1 Optimisation of reaction conditions[a]

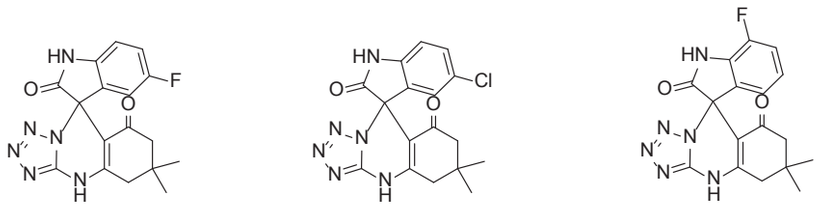


1 **2** **3** **4a**

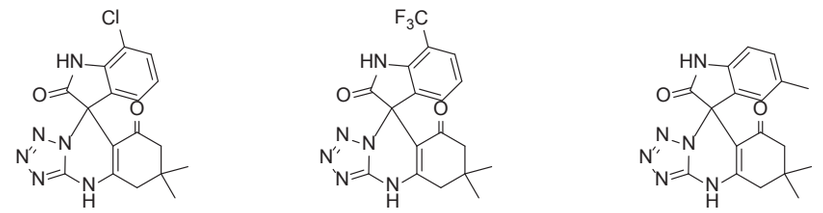
Entry	Solvent	Catalyst/mol%	Temperature/°C	Time/h	Yield/%[b]
1	CH_2Cl_2	$[MeC(OH)_2]^+ClO_4^-$ (10)	Reflux	12	15
2	Toluene	$[MeC(OH)_2]^+ClO_4^-$ (10)	Reflux	12	40
3	THF	$[MeC(OH)_2]^+ClO_4^-$ (10)	Reflux	12	42
4	EtOAc	$[MeC(OH)_2]^+ClO_4^-$ (10)	Reflux	12	38
5	MeOH	$[MeC(OH)_2]^+ClO_4^-$ (10)	Reflux	12	71
6	EtOH	$[MeC(OH)_2]^+ClO_4^-$ (10)	Reflux	6	82
7	H_2O	$[MeC(OH)_2]^+ClO_4^-$ (10)	Reflux	12	65
8	EtOH/H_2O (1:1)	$[MeC(OH)_2]^+ClO_4^-$ (10)	80	3	90
9	EtOH/H_2O (1:2)	$[MeC(OH)_2]^+ClO_4^-$ (10)	80	3	83
10	EtOH/H_2O (1:1)	–	80	24	trace
11	EtOH/H_2O (1:1)	$[MeC(OH)_2]^+ClO_4^-$ (5)	80	12	41
12	EtOH/H_2O (1:1)	$[MeC(OH)_2]^+ClO_4^-$ (20)	80	3	91
13	EtOH/H_2O (1:1)	Conc. H_2SO_4 (50)	80	12	35
14	EtOH/H_2O (1:1)	$HClO_4$ (50)	80	12	60
15	EtOH/H_2O (1:1)	CH_3COOH (50)	80	12	21
16	EtOH/H_2O (1:1)	*p*-TSA (50)	80	12	53

[a]Reaction conditions: isatin (1 mmol), 5 amino-1*H*-tetrazole (1 mmol), dimedone (1 mmol), solvent (4 mL), heating.
[b]Isolated yield.

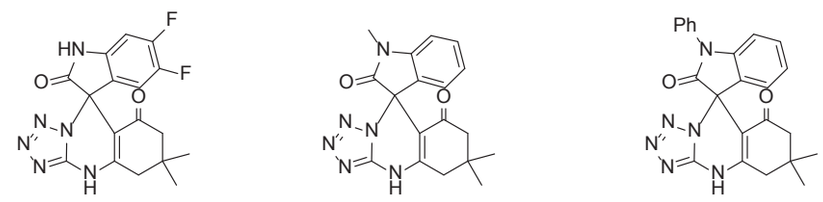
Table 2 Synthesis of spiroheterocycles **4a–j**[a]



4b: 92%[b] **4c: 88%**[b] **4d: 85%**[b]

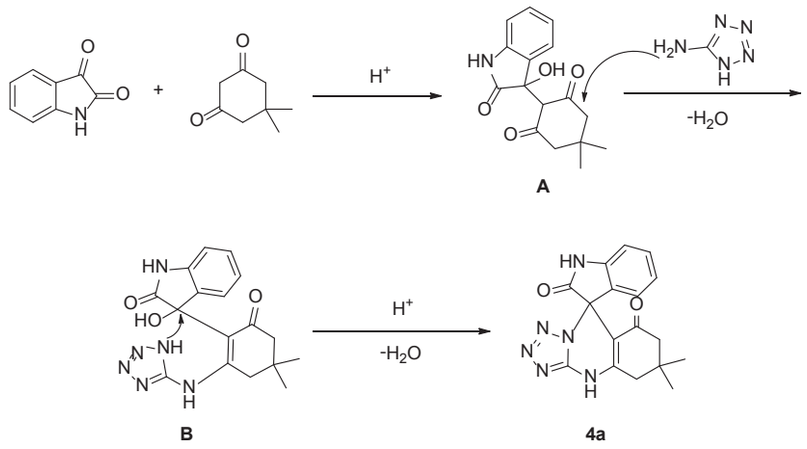


4e: 82%[b] **4f: 80%**[b] **4g: 87%**[b]



4h: 81%[b] **4i: 89%**[b] **4i: 86%**[b]

[a]Reaction conditions: substituted isatin (1 mmol), 5-amino-1*H*-tetrazole (1 mmol), dimedone (1 mmol), EtOH/H_2O (1:1, 4 mL), 80 °C, 3 h.
[b]Isolated yield.



Scheme 2 Plausible mechanism.

Experimental

Caution: Acetic acid–perchloric acid mixture is a super acid (pH = 4.4). The mixture can cause severe skin and eye burns. The vapour is irritating to the eyes and respiratory tract. Harmful if swallowed. Goggles and gloves must be worn.

All reagents were obtained from local commercial suppliers and used without further purification. Melting points were determined with a MPA100 apparatus (USA) and are uncorrected. ^{1}H and ^{13}C NMR spectra were recorded on a Bruker Avance III 400 analyser in DMSO-d_6 with TMS as internal standard. Mass spectra were taken on an Agilent 6540 UHD Accurate-Mass LC-MS instrument in the electrospray ionisation (positive ESI) mode.

Synthesis of the title compounds; typical procedure

A mixture of 5-amimotetrazole (1 mmol, 0.085 g), appropriate isatin (1 mmol), dimedone (1 mmol, 0.14 g) and $[MeC(OH)_2]^+ClO_4^-$ (10 mol%) was stirred in EtOH/H_2O (1:1, 4 mL) at 80 °C. After the indicated time, the reaction was quenched into water (50 mL) and cooled. The precipitated product was separated by centrifugation and purified by silica gel chromatography to afford the desired product. All the products were characterised by melting point, ^{1}H NMR, ^{13}C NMR and LC-MS.

6',6'-Dimethyl-6',7'-dihydro-4'H-spiro[indole-3,9'-tetrazolo[5,1-b]quinazoline]-2,8'(1H,5'H)-dione **(4a)**: Yield 90%; white solid, m.p. 312–316 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 12.03 (s, 1H), 10.88 (s, 1H), 7.28 (t, *J* = 7.6 Hz, 1H), 7.10 (d, *J* = 7.3 Hz, 1H), 6.91 (dd, *J* = 15.0, 7.6 Hz, 2H), 2.70 (d, *J* = 16.0 Hz, 1H), 2.62 (d, *J* = 16.0 Hz, 1H), 2.18 (d, *J* = 16.0 Hz, 1H), 2.12 (d, *J* = 16.0 Hz, 1H), 1.05 (d, *J* = 10.0 Hz, 6H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 193.14, 173.55, 153.15, 148.74, 143.02, 130.75, 130.16, 123.99, 122.66, 110.55, 104.92, 65.51, 50.23, 32.90, 28.23, 27.37; HRMS (ESI$^+$): calcd for $C_{17}H_{17}N_6O_2$: 337.1414, found $[M+H]^+$: 337.1411.

5-Fluoro-6',6'-dimethyl-6',7'-dihydro-4'H-spiro[indole-3,9'-tetrazolo[5,1-b]quinazoline]-2,8'(1H,5'H)-dione **(4b)**: Yield 90%; white solid, m.p. 306–310 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 12.08 (s, 1H), 10.92 (s, 1H), 7.21 (dd, *J* = 7.9, 2.2 Hz, 1H), 7.12 (s, 1H), 6.93 (d, *J* = 4.2 Hz, 1H), 2.69 (d, *J* = 16.0 Hz, 1H), 2.61 (d, *J* = 16.0 Hz, 1H), 2.18 (d, *J* = 10.9 Hz, 2H), 1.05 (s, 6H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 193.25, 173.68, 159.85, 157.49, 153.47, 148.69, 139.36, 131.44, 117.02, 112.45, 111.36, 104.57, 65.63, 50.19, 32.91, 27.84; HRMS (ESI$^+$): calcd for $C_{17}H_{16}FN_6O_2$: 355.1320, found $[M+H]^+$: 355.1318.

5-Chloro-6',6'-dimethyl-6',7'-dihydro-4'H-spiro[indole-3,9'-tetrazolo[5,1-b]quinazoline]-2,8'(1H,5'H)-dione **(4c)**: Yield 87%; white solid, m.p. 306–308 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 12.08 (s, 1H), 11.03 (s, 1H), 7.63–7.15 (m, 2H), 6.94 (d, *J* = 8.3 Hz, 1H), 2.68 (d, *J* = 16.0 Hz, 1H), 2.60 (d, *J* = 16.0 Hz, 1H), 2.23 (d, *J* = 16.0 Hz, 1H), 2.13 (d, *J* = 16.0 Hz, 1H), 1.05 (s, 6H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 193.33, 173.44, 153.62, 148.70, 142.05, 131.84, 130.59, 126.62, 124.63, 111.94, 104.46, 65.39, 50.17, 32.95, 27.84; HRMS (ESI$^+$): calcd for $C_{17}H_{16}ClN_6O_2$: 371.1024, found $[M+H]^+$: 371.1021.

7-Fluoro-6',6'-dimethyl-6',7'-dihydro-4'H-spiro[indole-3,9'-tetrazolo[5,1-b]quinazoline]-2,8'(1H,5'H)-dione **(4d)**: Yield 83%; white solid, m.p. 297–299 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 12.14 (s, 1H), 11.47 (s, 1H), 7.51–7.11 (m, 1H), 7.02 (d, *J* = 7.1 Hz, 1H), 6.98–6.66 (m, 1H), 2.72 (d, *J* = 16.0 Hz, 1H), 2.62 (d, *J* = 16.0 Hz, 1H), 2.23 (d, *J* = 16.0 Hz, 1H), 2.13 (d, *J* = 16.0 Hz, 1H), 1.05 (d, *J* = 11.1 Hz, 6H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 193.34, 173.38, 153.46, 148.65, 145.67, 132.63, 130.18, 123.67, 120.25, 117.93, 104.66, 65.40, 50.10, 32.94, 28.18, 27.38; HRMS (ESI$^+$): calcd for $C_{17}H_{16}FN_6O_2$: 355.1320, found $[M+H]^+$:355.1315.

7-Chloro-6',6'-dimethyl-6',7'-dihydro-4'H-spiro[indole-3,9'-tetrazolo[5,1-b]quinazoline]-2,8'(1H,5'H)-dione **(4e)**: Yield 79%; white solid, m.p. 296–298 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 12.15 (s, 1H), 11.38 (s, 1H), 7.37 (dd, *J* = 8.2, 1.0 Hz, 1H), 7.15 (d, *J* = 7.2 Hz, 1H), 7.03–6.84 (m, 1H), 2.72 (d, *J* = 16.0 Hz, 1H), 2.63 (d, *J* = 16.0 Hz, 1H), 2.24 (d, *J* = 16.0 Hz, 1H), 2.14 (d, *J* = 16.0 Hz, 1H), 1.05 (d, *J* = 11.3 Hz, 6H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 193.35, 173.59, 153.51, 148.64, 140.82, 131.68, 130.80, 124.04, 122.86, 114.80, 104.65, 65.88, 50.09, 32.95, 28.18, 27.39; HRMS (ESI$^+$): calcd for $C_{17}H_{16}ClN_6O_2$: 371.1024, found $[M+H]^+$: 371.1021.

6',6'-Dimethyl-7-(trifluoromethyl)-6',7'-dihydro-4'H-spiro[indole-3,9'-tetrazolo[5,1-b]quinazoline]-2,8'(1H,5'H)-dione **(4f)**: Yield 89%; white solid, m.p. 302–304 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 12.01 (s, 1H), 10.78 (s, 1H), 7.07 (dd, *J* = 7.9, 0.9 Hz, 1H), 6.91 (s, 1H), 6.81 (d, *J* = 7.9 Hz, 1H), 2.66 (d, *J* = 3.6 Hz, 2H), 2.36–2.02 (m, 5H), 1.05 (d, *J* = 4.0 Hz, 6H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 193.36, 174.12, 153.81, 148.76, 140.51, 131.73, 128.27, 127.25, 125.29, 122.96, 112.68, 111.37, 104.50, 64.27, 50.04, 32.96, 28.13, 27.44; HRMS (ESI$^+$): calcd for $C_{18}H_{16}F_3N_6O_2$: 405.1288, found $[M+H]^+$: 405.1283.

5,6',6'-Trimethyl-6',7'-dihydro-4'H-spiro[indole-3,9'-tetrazolo[5,1-b]quinazoline]-2,8'(1H,5'H)-dione **(4g)**: Yield 77%; white solid, m.p. 280–284 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 12.01 (s, 1H), 10.78 (s, 1H), 7.07 (dd, *J* = 7.9, 0.9 Hz, 1H), 6.91 (s, 1H), 6.81 (d, *J* = 7.9 Hz, 1H), 2.66 (d, *J* = 3.6 Hz, 2H), 2.36–2.02 (m, 5H), 1.05 (d, *J* = 4.0 Hz, 6H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 193.15, 173.52, 153.01, 148.69, 140.56, 131.70, 130.96, 130.24, 124.46, 110.30, 104.97, 65.63, 50.25, 32.93, 28.07, 27.60, 20.93; HRMS (ESI$^+$): calcd for $C_{18}H_{19}N_6O_2$: 351.1570, found $[M+H]^+$: 351.1570.

5,6-Difluoro-6',6'-dimethyl-6',7'-dihydro-4'H-spiro[indole-3,9'-tetrazolo[5,1-b]quinazoline]-2,8'(1H,5'H)-dione **(4h)**: Yield 75%; white solid, m.p. 330–332 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 12.09 (s, 1H), 11.05 (s, 1H), 7.51 (dd, *J* = 9.7, 8.0 Hz, 1H), 7.00 (dd, *J* = 10.4, 6.6 Hz, 1H), 2.69 (d, *J* = 16.0 Hz, 1H), 2.61 (d, *J* = 16.0 Hz, 1H), 2.32–2.07 (m, 2H), 1.05 (s, 6H); ^{13}C NMR (100 MHz, DMSO-*d*6) δ 193.32, 173.80, 153.65, 148.70, 139.99, 125.81, 114.60, 114.39, 104.38, 100.46, 100.19,

65.22, 50.16, 32.91, 28.06, 27.63; HRMS (ESI$^+$): calcd for $C_{17}H_{15}F_2N_6O_2$: 373.1225, found $[M+H]^+$: 373.1223.

1,6',6'-Trimethyl-6',7'-dihydro-4'H-spiro[indole-3,9'-tetrazolo[5,1-b]quinazoline]-2,8'(1H,5'H)-dione **(4i)**: Yield 88%; white solid, m.p. 332–334 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 12.10 (s, 1H), 7.39 (td, *J*=7.7, 1.2 Hz, 1H), 7.27–7.09 (m, 2H), 6.99 (t, *J*=7.5 Hz, 1H), 3.25 (s, 3H), 2.75 (d, *J*=16.0 Hz, 1H), 2.67 (d, *J*=16.0 Hz, 1H), 2.19 (d, *J*=16.0 Hz, 1H), 2.11 (d, *J*=16.0 Hz, 1H), 1.05 (d, *J*=10.2 Hz, 6H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 193.16, 172.23, 153.26, 148.80, 144.39, 130.93, 129.37, 123.73, 123.40, 109.47, 104.89, 65.03, 50.18, 32.93, 28.15, 27.43, 27.14; HRMS (ESI$^+$): calcd for $C_{18}H_{19}N_6O_2$: 351.1570, found $[M+H]^+$: 351.1567.

6',6'-Dimethyl-1-phenyl-6',7'-dihydro-4'H-spiro[indole-3,9'-tetrazolo[5,1-b]quinazoline]-2,8'(1H,5'H)-dione **(4j)**: Yield 87%; white solid, m.p. 325–327 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 12.22 (s, 1H), 7.65 (t, *J*=7.7 Hz, 2H), 7.53 (t, *J*=7.5 Hz, 3H), 7.32 (dd, *J*=14.2, 7.4 Hz, 2H), 7.05 (t, *J*=7.5 Hz, 1H), 6.81 (d, *J*=7.9 Hz, 1H), 2.75 (d, *J*=16.0 Hz, 1H), 2.67 (d, *J*=16.0 Hz, 1H), 2.28 (d, *J*=16.0 Hz, 1H), 2.18 (d, *J*=16.0 Hz, 1H), 1.07 (d, *J*=6.1 Hz, 6H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 193.60, 171.85, 153.49, 148.88, 144.22, 134.61, 131.08, 130.34, 129.01, 127.04, 124.45, 124.07, 109.87, 105.23, 65.25, 50.07, 33.01, 28.25, 27.40; HRMS (ESI$^+$): calcd for $C_{23}H_{21}N_6O_2$: 413.1727, found $[M+H]^+$: 413.1724.

Conclusions

We have described a superacid catalysed highly efficient, one-pot protocol for the synthesis of spiro[indole-3,9'-tetrazolo[5,1-*b*]quinazoline]-2,8'(1*H*,5'H)-diones in good to excellent yields in aqueous medium. Easy work up, the ready availability of the catalyst, mild conditions and an environmentally friendly reaction medium make the procedure attractive and acceptable.

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