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Expedient synthesis of novel coumarin-based sulfonamides

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Abstract The synthesis of novel coumarin-based sulfonamides was accomplished in good yields via $InCl_3$-catalyzed three-component condensation of *N*-(2-formylphenyl)-*N*-methylbenzenesulfonamides, 4-hydroxycoumarin and cyclic secondary amines in toluene.

Keywords Coumarin-based sulfonamides · Sulfonamides · Coumarins · Multicomponent

Introduction

The so-called multicomponent reactions (MCRs) are one-pot processes in which at least three or more different simple substrates react for the preparation of target materials [1–5]. These reactions which have gained much attention during the past years are frequently occurring not through a single-step procedure, but rather by several sequential steps or multicomponent cascade or domino reactions [6–10]. Simplicity, greater efficiency and atom economy with generation of molecular complexity and diversity in one-pot transformation are some of the advantages of these reactions.

Sulfonamides, described as privileged structures [11], have a rich chemical and biological history and are an important class of compounds in drug discovery due to their extensive chemical and biological activities [12, 13]. Sulfonamides have been used extensively as antibacterial [14], anti-carbonic anhydrase [15], diuretic [16] and hypoglycemic reagents [17]. As pharmaceutical agents, they have been used for treatment of infections [18], Alzheimer's disease [19], HIV [20] and cancer [21].

The known properties of compounds containing the sulfonamide moieties along with the documented multiple biological activities of coumarins in treating various cancer, cardiovascular, and rheumatic diseases [22–30] prompted us to prepare novel coumarin-based sulfonamides since significant enhancement in biological activity in the presence of two different motifs in a single molecule was expected [31].

Experimental

General information

All commercially available chemicals and reagents were used without further purification. Melting points were determined with an electrothermal mode 19100 apparatus and were uncorrected. IR spectra were recorded on a Shimadzu 4300 spectrophotometer, in cm^{-1}. ^1H and ^{13}C-NMR Spectra: Bruker AVANCE 300 spectrometer at 300 (^1H) and 75 MHz (^{13}C) and Bruker Ultrashield 400 spectrometer at 400 (^1H) and 100 MHz (^{13}C) using $CDCl_3$ as solvent and with the residual solvent signal as internal reference ($CDCl_3$, 7.24 ppm). Mass spectra of the products were obtained with a HP (Agilent technologies) 5937 mass selective detector. Elemental analyses were carried out by a CHN-Rapid Heraeus elemental analyzer (Wellesley, MA).

*General procedure for the preparation of **3c–e***

To a stirred solution containing *N*-methylaminobenzaldehydes (10 mmol) and pyridine (15 mmol), dry CH_2Cl_2 (15 mL) was added dropwise to a solution of

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p-toluenesulfonyl chloride (12 mmol). The mixture was stirred at 25 ºC for 48 h. The organic solution was then washed with water (30 mL) and saturated sodium sulfite solution (30 mL), and dried over Na_2SO_4. After removal of the solvent under reduced pressure, the crude residue was purified by column chromatography on silica gel (230–400 mesh; Merck), using hexane–EtOAc (5:1, 3:1 and 1:1) as eluent to give **3c–e**.

*N-(2-Formyl-4-methoxyphenyl)-N,4-dimethylbenzenesulfonamide (**3c**)* Yellow solid, mp: 106–108 ºC; yield: 0.220 g (69 %). IR (KBr) (υ_{max}, cm^{-1}): 1,682, 1,332, 1,151; ^{1}H NMR (500 MHz, $CDCl_3$): $\delta_H = 2.45$ (s, 3H, Me), 3.21 (s, 3H, NMe), 3.86 (s, 3H, OMe), 6.59 (d, $J = 8.8$ Hz, 1H, Ar), 6.98 (dd, $J = 8.8$, 2.6 Hz, 1H, Ar), 7.29 (d, $J = 8.0$ Hz, 2H, Ar), 7.44–7.47 (m, 3H, Ar), 10.41 (s, 1H, CHO); ^{13}C NMR (125 MHz, $CDCl_3$): δ_C = 21.6, 39.1, 55.7, 110.6, 121.5, 127.4, 128.2 (2C), 129.6 (2C), 132.9, 135.9, 136.6, 144.2, 159.1, 189.9 (CO); EI-MS: *m/z* (%): 319 (8, M $^+$), 164 (100), 155 (8), 136 (86), 91 (65); Anal. calcd for $C_{16}H_{17}NO_4S$ (319.09): C 60.17, H 5.37, N 4.39 %. Found: C 60.23, H 5.46, N 4.50 %.

*N-(2-Formylphenyl)-N,4-dimethylbenzenesulfonamide (**3d**)* White solid, mp: 137–139 ºC; yield: 0.159 g (55 %). IR (KBr) (υ_{max}, cm^{-1}): 1,682 (CO), 1,345, 1,151; ^{1}H NMR (500 MHz, $CDCl_3$): $\delta_H = 2.44$ (s, 3H, Me), 3.23 (s, 3H, NMe), 6.69 (d, $J = 7.8$ Hz, 1H, Ar), 7.28 (d, $J = 8.0$ Hz, 2H, Ar), 7.41–7.48 (m, 4H, Ar), 8.00 (dd, $J = 7.5$, 2.4 Hz, 1H, Ar), 10.46 (s, 1H, CHO); ^{13}C NMR (125 MHz, $CDCl_3$): δ_C = 21.6, 38.8, 126.0, 128.2 (2C), 128.3, 128.3, 129.6 (2C), 132.6, 134.0, 134.9, 143.7, 144.3, 189.9 (CO); EI-MS: *m/z* (%) : 289 (2, M $^+$), 196 (5), 155 (10), 134 (100), 91 (85); Anal. calcd for $C_{15}H_{15}NO_3S$ (289.08): C 62.27, H 5.23, N 4.84 %. Found: C 62.30, H 5.44, N 5.01 %.

*N-Ethyl-N-(2-formylphenyl)-4-methylbenzenesulfonamide (**3e**)* White solid, mp: 105–107 ºC, yield: 0.152 g (50 %). IR (KBr) (υ_{max}, cm^{-1}): 1,690 (CO), 1,346, 1,152; ^{1}H NMR (500 MHz, $CDCl_3$): $\delta_H = 1.10$ (t, $J = 9.5$ Hz, 3H, CH_2CH_3), 2.44 (s, 3H, Me), 3.33 (bs, 1H, NCH), 4.01 (bs, NCH), 6.71 (d, $J = 7.8$ Hz, 1H, Ar), 7.27 (d, $J = 7.7$ Hz, 2H, Ar), 7.42–7.48 (m, 4H, Ar), 8.02 (dd, $J = 7.6$, 2.4 Hz, 1H, Ar), 10.44 (s, 1H, CHO); ^{13}C NMR (125 MHz, $CDCl_3$): δ_C = 13.8, 21.6, 46.3, 127.5, 127.9 (2C), 128.4, 128.5, 129.6 (2C), 134.1, 134.3, 136.4, 141.3, 144.0, 190.2 (CO); EI-MS: *m/z* (%): 303 (1, M $^+$), 210 (1), 155 (8), 148 (100), 91 (37). Anal. calcd for $C_{16}H_{17}NO_3S$ (303.09): C 63.35, H 5.65, N 4.62 %. Found: C 63.52, H 5.86, N 4.66 %.

Typical procedure for the preparation of **4a–n**. To a solution of an aldehyde (1 mmol), 4-hydroxycoumarin (162 mg, 1 mmol) and secondary amine (1 mmol) in toluene (5 mL), $InCl_3$ was added (10 mol %) and the mixture was stirred for 3 h at room temperature. After evaporation of the solvent at reduced pressure and addition of 10 mL of *n*-hexane, the solid was separated by filtration. Recrystallization of the solid from ethyl acetate finally afforded coumarin-based sulfonamides **4a–n**.

*(E)-N-(2-((4-Hydroxy-2-oxo-2H-chromen-3-yl)(pyrrolidin-1-yl)methyl)phenyl)-N-methyl-2-phenylethenesulfonamide (**4a**)* White solid, mp: 179–181 ºC, yield: 0.366 g (71 %). IR (KBr) (υ_{max}, cm^{-1}): 2,983, 1,636, 1,454, 1,343, 1,140; ^{1}H-NMR (400 MHz, $CDCl_3$): $\delta_H = 2.02$–2.10 (m, 2H, CH_2), 2.16–2.22 (m, 2H, CH_2), 3.14–3.34 (m, 2H, NCH_2), 3.44 (s, 3H, NMe), 3.48–3.70 (m, 2H, NCH_2), 6.11 (*s*, 1H, NCH), 7.00 (d, $J = 15.5$ Hz, 1H, CH), 7.20 (*d*, $J = 8.2$ Hz, 1H, Ar), 7.24 (m, 1H, Ar), 7.28 (*t*, $J = 2.0$ Hz, 1H, Ar), 7.34–7.37 (m, 2H, Ar), 7.43–7.50 (m, 5H, Ar), 7.57–7.61 (m, 2H, Ar), 8.01 (dd, $J = 7.8$, 1.7 Hz, 1H, Ar), 8.25 (d, $J = 9.7$ Hz, 1H, Ar), 14.65 (bs, 1H, OH); ^{13}C NMR (125 MHz, $CDCl_3$): δ_C = 23.9, 24.4, 41.2, 50.5, 55.1, 64.5, 95.5, 113.8, 115.9, 116.4, 121.4, 121.9, 122.9, 124.2, 124.4, 128.4 (2C), 128.5, 129.2 (2C), 131.2, 131.3, 132.6, 133.1, 141.2, 143.8, 154.0, 164.0; 174.8; EI-MS: *m/z* (%): 516 (3, M $^+$), 445 (56), 381 (54), 304 (82), 220 (100). Anal. calcd for $C_{29}H_{28}N_2O_5S$ (516.17): C 67.42, H 5.46, N 5.42 %. Found: C 67.12, H 5.57, N 5.66 %.

*(E)-N-(2-((4-Hydroxy-2-oxo-2H-chromen-3-yl)(piperidin-1-yl)methyl)phenyl)-N-methyl-2-phenylethene-1-sulfonamide (**4b**)* White solid, mp: 190–192 ºC, yield: 0.345 g (65 %). IR (KBr) (υ_{max}, cm^{-1}): 3,065, 1,638, 1,452, 1,333, 1,135; ^{1}H-NMR (400 MHz, $CDCl_3$): δ_H = 1.60–1.68 (m, 2H, CH_2), 1.82–1.88 (m, 4H, $2CH_2$), 2.85-2.90 (m, 1H, NCH), 2.93–3.00 (m, 1H, NCH), 3.04-3.10 (m, 1H, NCH), 3.39 (s, 3H, NMe), 3.78-3.83 (m, 1H, NCH), 5.86 (s, 1H, CHN), 6.88 (d, $J = 15.5$ Hz, 1H, SO_2CH), 7.05 (d, $J = 8.0$ Hz, 1H, Ar), 7.12–7.15 (m, 2H, Ar), 7.25 (t, $J = 4.8$ Hz, 1H, Ar), 7.32 (t, $J = 4.6$ Hz, 1H, Ar), 7.37–7.40 (m, 5H, Ar), 7.48–7.52 (m, 2H, Ar), 7.93 (dd, $J = 7.8$, 1.5 Hz, 1H, Ar), 8.02 (d, $J = 9.5$ Hz, 1H, Ar), 14.2 (bs, 1H, OH); ^{13}C NMR (125 MHz, $CDCl_3$): δ_C = 22.5, 24.8, 24.9, 41.1, 50.0, 54.2, 65.2, 95.2, 115.8, 116.5, 116.8, 120.8, 121.8, 122.9, 123.2, 124.3, 128.4 (2C), 128.6, 129.3 (2C), 130.6, 131.3, 132.5, 132.9, 137.2, 144.0, 154.0, 164.2, 174.8; EI-MS: *m/z* (%): 530 (4, M^+), 445 (34), 429 (1), 262 (29), 91 (100). Anal. calcd for $C_{30}H_{30}N_2O_5S$ (530.19): C 67.90, H 5.70, N 5.28 %. Found: C 68.30, H 5.92, N 5.29 %.

*tert-Butyl (E)-4-((4-hydroxy-2-oxo-2H-chromen-3-yl)(2-((N-methyl-2-phenylvinyl)sulfonamido)phenyl)methyl)piperazine-1-carboxylate (**4c**)* White solid, mp: 216–218 ºC, yield : 0.372 g (59 %). IR (KBr) (υ_{max}, cm^{-1}): 2,979, 1,683, 1,450, 1,334, 1,139; ^{1}H-NMR (400 MHz, $CDCl_3$): $\delta_H = 1.49$ (s, 9H, CMe_3), 2.64 (s, 3H, NMe), 3.44–3.47

(m, 4H, $2NCH_2$), 3.76–3.79 (m, 4H, 2 NCH_2), 6.65 (s, 1H, CHN), 6.83 (d, $J = 15.5$ Hz, 1H, SO_2CH), 7.02 (dd, $J = 7.8$, 1.4 Hz, 1H, Ar), 7.17–7.20 (m, 2H, Ar), 7.29–7.32 (m, 2H, Ar), 7.37 (d, $J = 15.5$ Hz, 1H, CH =), 7.40–7.46 (m, 5H, Ar), 7.51 (m, 1H, Ar), 7.63 (d, $J = 7.8$ Hz, 1H, Ar), 7.98 (dd, $J = 7.9$, 1.6 Hz, 1H, Ar), 8.17 (d, $J = 8.1$ Hz, 1H, Ar), 8.70 (bs, 1H, OH); ^{13}C NMR (125 MHz, $CDCl_3$): $\delta_C = 28.3$ (3C), 35.0 (2C), 38.7, 44.4 (2C), 81.1, 100.7, 107.3, 115.8, 120.5, 123.3, 123.5, 125.0, 127.1, 127.1, 128.3 (2C), 128.8, 129.2 (2C), 130.5, 130.9, 131.2, 131.4, 132.4, 142.2, 143.7, 152.7, 166.9, 169.6, 172.0; EI-MS: *m/z* (%): 631 (5, M^+), 430 (1), 351 (20), 206 (100), 77 (28); Anal. calcd for $C_{34}H_{37}N_3O_7S$ (631.24): C 64.64, H 5.90, N 6.65 %. Found: C 64.83, H 5.67, N 6.77 %.

(E)-N-(2-((4-Hydroxy-2-oxo-2H-chromen-3-yl)(pyrrolidin-1-yl)methyl)-4-methoxyphenyl)-N-methyl-2-phenylethene-1-sulfonamide ***(4d)*** White solid, mp: 189–191 ºC, yield: 0.377 g (69 %). IR (KBr) (υ_{max}, cm^{-1}): 2,989, 1,640, 1,464, 1,313, 1,132; ^{1}H-NMR (400 MHz, $CDCl_3$): δ_H = 1.27–1.34 (m, 2H, CH_2), 1.91–1.99 (m, 2H, CH_2), 3.13–3.25 (m, 2H, NCH_2), 3.32 (s, 3H, NMe), 3.53–3.60 (m, 2H, NCH_2), 3.68 (s, 3H, OMe), 5.96 (s, 1H, NCH), 6.77 (dd, $J = 8.8$, 3.0 Hz, 1H, Ar), 6.88 (d, $J = 15.5$, 1H, SO_2CH), 7.09 (d, $J = 8.8$ Hz, 1H, Ar), 7.13 (t, $J = 7.5$ Hz, 1H, Ar), 7.20 (s, 1H, Ar), 7.35–7.41 (m, 5H, Ar), 7.48 (d, $J = 15.5$ Hz, 1H, CH =), 7.50 (t, $J = 2.6$ Hz, 1H, Ar), 7.73 (d, $J = 2.8$ Hz, 1H, Ar), 7.91 (dd, $J = 7.9$, 1.7 Hz, 1H, Ar), 14.42 (bs,1H, OH); ^{13}C NMR (125 MHz, $CDCl_3$): δ_C = 23.9, 24.4, 41.2, 50.5, 55.1, 55.6, 64.5, 95.5, 113.8, 115.9, 116.4, 121.4, 121.9, 122.9, 124.2, 128.4, 128.5, 129.2 (2C), 131.2 (2C), 131.3, 132.6, 133.1, 140.2, 143.8, 154.0, 159.9, 164.0, 174.8; EI-MS: *m/z* (%): 546 (2, M^+), 475 (16), 430 (1), 334 (16), 188 (19), 70 (100). Anal. calcd for $C_{30}H_{30}N_2O_6S$ (546.18): C 65.92, H 5.53, N 5.12 %. Found: C 65.84, H 5.53, N 5.26 %.

(E)-N-(2-((4-Hydroxy-2-oxo-2H-chromen-3-yl)(piperidin-1-yl)methyl)-4-methoxyphenyl)-N-methyl-2-phenylethene-1-sulfonamide ***(4e)*** White solid, mp: 198–200 ºC, yield: 0.336 g (60 %). IR (KBr) (υ_{max}, cm^{-1}): 2,952, 1,670, 1,454, 1,333, 1,136; ^{1}H-NMR (400 MHz, $CDCl_3$): δ_H = 1.72–1.80 (m, 2H, CH_2), 1.93–1.98 (m, 4H, $2CH_2$), 2.94–3.02 (m, 1H, NCH), 3.09–3.16 (m, 1H, NCH), 3.24–3.28 (m, 1H, NCH), 3.45 (s, 3H, NMe), 3.75 (s, 3H, OMe), 3.87–3.90 (m, 1H, NCH), 5.91 (s, 1H, NCH), 6.87 (dd, $J = 8.6$, 3.0 Hz, 1H, Ar), 6.96 (d, $J = 15.4$ Hz, 1H, SO_2CH), 7.19 (d, $J = 8.9$ Hz, 1H, Ar), 7.20 (t, $J = 8.5$ Hz, 1H, Ar), 7.28 (s, 1H, Ar), 7.36–7.50 (m, 5H, Ar), 7.58 (d, $J = 15.4$ Hz, 1H, CH=), 7.57 (t, $J = 7.6$ Hz, 1H, Ar), 7.70 (d, $J = 3.0$ Hz, 1H, Ar), 8.02 (dd, $J = 7.8$, 1.4, 1.0 Hz, 1H, Ar), 14.15 (bs, 1H, OH); ^{13}C NMR (125 MHz, $CDCl_3$): δ_C = 22.5, 24.9, 24.9, 41.2, 50.0, 54.2, 55.6, 65.3, 95.0, 114.4, 116.1, 116.5, 120.8, 121.9, 122.9, 124.3, 128.4, 128.5 (2C), 129.2 (2C), 131.2, 131.3,132.5, 133.7, 138.5, 143.8, 154.0, 159.8, 164.2, 174.9; EI-MS: *m/z* (%): 560 (2, M^+), 475 (100), 413 (3), 292 (33), 103 (32); Anal. calcd for $C_{31}H_{32}N_2O_6S$ (560.20): C 66.41, H 5.75, N 5.00 %. Found: C 66.04, H 6.10, N 5.11 %.

tert-Butyl (E)-4-((4-hydroxy-2-oxo-2H-chromen-3-yl)(5-methoxy-2-((N-methyl-2-phenylvinyl)sulfonamido)phenyl)methyl)piperazine-1-carboxylate ***(4f)*** White solid, mp: 219–221 ºC, yield: 0.370 g (56 %). IR (KBr) (υ_{max}, cm^{-1}): 2,978, 1,683, 1,454, 1,337, 1,155; ^{1}H-NMR (400 MHz, $CDCl_3$): δ_H = 1.41 (s, 9H, CMe_3), 2.53 (s, 3H, NMe), 3.39 (t, $J = 5.2$ Hz, 4H, $2NCH_2$), 3.64 (s, 3H, OMe), 3.74 (t, $J = 5.1$ Hz, 4H, $2NCH_2$), 6.49 (s, 1H, NCH) 6.58 (dd, $J = 8.6$, 3.0 Hz, 1H, Ar), 6.75 (d, $J = 15.5$, 1H, SO_2CH), 6.84 (d, $J = 8.8$ Hz, 1H, Ar), 7.11 (t, $J = 8.8$ Hz, 1H, Ar), 7.19 (s, 1H, Ar), 7.28 (d, $J = 15.5$ Hz, 1H, CH =), 7.36–7.40 (m, 5H, Ar), 7.49 (t, $J = 3.9$ Hz, 1H, Ar), 7.91 (dd, $J = 7.8$, 1.6 Hz, 1H, Ar), 8.08 (dd, $J = 7.9$, 1.7 Hz, 1H, Ar), 8.57 (bs, 1H, OH); ^{13}C NMR (125 MHz, $CDCl_3$): δ_C = 28.3 (3C), 35.1 (2C), 38.7, 44.6 (2C), 55.2, 81.2, 100.7, 107.0, 111.9, 115.8, 115.9, 120.4, 123.3, 123.5, 125.0, 128.3, 128.4 (2C), 129.2 (2C), 130.9, 131.2, 131.4, 132.4, 141.0, 143.6, 154.0, 159.3, 163.6, 169.9, 172.0; EI-MS: *m/z* (%): 661 (2, M^+), 475 (35), 411 (6), 380 (2), 334 (32), 57 (100); Anal. calcd for $C_{35}H_{39}N_3O_8S$ (661.25): C 63.52, H 5.94, N 6.35 %. Found: C 63.44, H 6.13, N 6.23 %.

N-(2-((4-Hydroxy-2-oxo-2H-chromen-3-yl)(pyrrolidin-1-yl)methyl)phenyl)-N,4-dimethylbenzenesulfonamide ***(4g)*** White solid, mp : 209–211 ºC, yield 0.398 g (79 %). IR (KBr) (υ_{max}, cm^{-1}): 3,085, 1,673, 1,461, 1,335, 1,156; ^{1}H-NMR (400 MHz, $CDCl_3$): δ_H = 1.88–2.01 (m, 2H, CH_2), 2.10–2.18 (m, 2H, CH_2), 2.42 (s, 3H, Me), 3.06–3.28 (m, 2H, NCH_2), 3.29 (s, 3H, NMe), 3.48–3.63 (m, 2H, NCH_2), 6.07 (s, 1H, NCH), 6.42 (d, $J = 8.0$ Hz, 1H, Ar), 7.06 (m, 1H, Ar), 7.11 (d, $J = 8.2$ Hz, 1H, Ar), 7.15 (t, $J = 7.4$ Hz, 1H, Ar), 7.22 (t, $J = 7.5$ Hz, 1H, Ar), 7.29 (d, $J = 8.1$ Hz, 2H, Ar), 7.36 (m, 1H, Ar), 7.59 (d, $J = 8.1$ Hz, 2H, Ar), 7.91 (dd, $J = 7.8$,1.5 Hz, 1H, Ar), 8.13 (dd, $J = 8.0$, 1.3 Hz, 1H, Ar), 14.92 (bs, 1H, OH); ^{13}C NMR (125 MHz, $CDCl_3$): δ_C = 21.7, 24.0, 24.4, 41.4, 50.2, 55.2, 64.4, 96.0, 116.4, 121.3, 122.9, 124.1, 127.0, 128.5 (2C), 129.4, 129.5, 129.6, 129.7 (2C), 131.2, 134.1, 139.2, 140.6, 144.1, 154.0, 163.3, 174.5; EI-MS: *m/z* (%): 504 (2, M^+), 347 (70), 280 (100), 249 (58), 139 (4), 91 (80). Anal. calcd for $C_{28}H_{28}N_2O_5S$ (504.17): C 66.65, H 5.59, N 5.55 %. Found: C 66.65, H 5.47, N 5.71 %.

N-(2-((4-Hydroxy-2-oxo-2H-chromen-3-yl)(piperidin-1-yl)methyl)phenyl)-N,4-dimethylbenzenesulfonamide ***(4h)*** White solid, mp: 238–240 ºC, yield: 0.383 g (74 %). IR (KBr) (υ_{max}, cm^{-1}): 2,955, 1,687, 1,450, 1,338, 1,171;

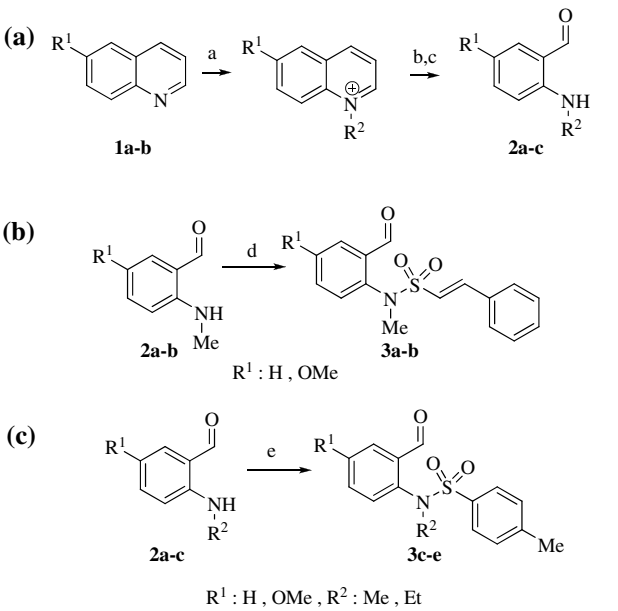
^{1}H-NMR (400 MHz, $CDCl_3$): $\delta_H = 1.66–1.72$ (m, 2H, CH_2), 1.87–1.93 (m, 4H, $2CH_2$), 2.44 (s, 3H, Me), 2.57 (s, 3H, OMe), 3.45 (t, $J = 4.9$ Hz, 4H, NCH_2), 6.22 (d, $J = 7.7$ Hz, 1H, Ar), 6.72 (s, 1H, NCH), 6.99 (t, $J = 7.4$ Hz, 1H, Ar), 7.16 (t, $J = 7.6$ Hz, 1H, Ar), 7.28 (d, $J = 7.5$ Hz, 2H, Ar), 7.41 (m, 1H, Ar), 7.49 (m, 3H, Ar), 7.52 (d, $J = 8.0$ Hz, 1H, Ar), 7.64 (d, $J = 7.9$ Hz, 1H, Ar), 8.15–8.18 (m, 2H, Ar, OH); ^{13}C NMR (125 MHz, $CDCl_3$): $\delta_C = 21.6$, 22.5, 23.1 (2C), 38.7, 45.8 (2C), 66.3, 100.8, 115.8, 120.8, 123.1, 124.9, 126.4, 126.6, 128.4, 128.5 (2C), 129.4 (2C), 130.3, 130.6, 131.2, 133.4, 139.3, 143.9, 152.8, 166.7, 171.8; EI-MS: *m/z* (%): 519 (2, M^++1), 278 (7), 249 (14), 162 (89), 120 (100), 92 (100). Anal. calcd for $C_{29}H_{30}N_2O_5S$ (518.19): C 67.16, H 5.83, N 5.40 %. Found: C 66.99, H 6.05, N 5.34 %.

tert-Butyl 4-((2-(N,4-dimethylphenylsulfonamido)phenyl)(2,4-dioxochroman-3-yl)methyl)piperazine-1-carboxylate ***(4i)*** White solid, mp: 252–254 ºC, yield: 0.427 g (69 %). IR (KBr) (υ_{max}, cm^{-1}): 2,977, 1,682, 1,453, 1,339, 1,153; ^{1}H-NMR (400 MHz, $CDCl_3$): $\delta_H = 1.48$ (s, 9H, CMe_3), 2.45 (s, 3H, Me), 2.56 (s, 3H, NMe), 3.46 (t, $J = 5.3$ Hz, 4H, $2NCH_2$), 3.74 (t, $J = 5.3$ Hz, 4H, $2NCH_2$), 6.23 (d, $J = 7.6$ Hz, 1H, Ar), 6.72 (s, 1H, NCH), 7.01 (t, $J = 7.4$ Hz, 1H, Ar), 7.17 (t, $J = 7.5$ Hz, 1H, Ar), 7.29 (d, $J = 6.9$ Hz, 2H, Ar), 7.43 (t, $J = 7.8$ Hz, 1H, Ar), 7.47-7.51 (m, 3H, Ar), 7.62 (d, $J = 7.8$ Hz, 1H, Ar), 7.97 (dd, $J = 7.9$, 1.6 Hz, 1H, Ar), 8.17 (d, $J = 7.6$ Hz, 1H, Ar), 8.76 (bs, 1H, OH); ^{13}C NMR (125 MHz, $CDCl_3$): $\delta_C = 21.6$, 28.3 (3C), 35.0 (2C), 38.7, 44.4 (2C), 81.0, 100.7, 107.5, 115.8, 120.6, 123.3, 125.0, 126.5, 126.7, 128.4, 128.6, 129.5, 130.2, 130.8, 131.3, 133.2, 142.2, 144.0, 152.8, 166.8, 169.3, 172.2; EI-MS: *m/z* (%): 619 (4, M^+), 241 (2), 197 (100), 90 (41),57 (50); Anal. calcd for $C_{33}H_{37}N_3O_7S$ (619.24): C 63.46, H 5.82, N 6.94 %. Found: C 63.75, H 5.96, N 7.04 %.

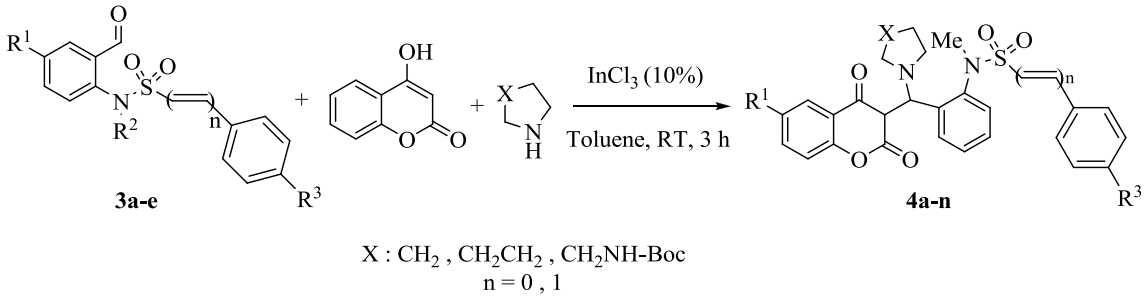
N-(2-((4-Hydroxy-2-oxo-2H-chromen-3-yl)(pyrrolidin-1-yl)methyl)-4-methoxyphenyl)-N,4-dimethylbenzenesulfonamide ***(4j)*** Yield 427 mg (80 %). White solid, mp: 204–206 ºC. IR (KBr) (υ_{max}, cm^{-1}): 3,110, 1,671, 1,460, 1,338, 1,155; ^{1}H-NMR (400 MHz, $CDCl_3$): $\delta_H = 1.98–2.07$ (m, 2H, CH_2), 2.12–2.27 (m, 2H, CH_2), 2.48 (s, 3H, Me), 3.17–3.28 (m, 2H, NCH_2), 3.33 (s, 3H, NMe), 3.57–3.68 (m, 2H, NCH_2), 3.72 (s, 3H, OMe), 6.10 (s, 1H, NCH), 6.38 (d, $J = 8.9$ Hz, 1H, Ar), 6.65 (dd, $J = 8.9$, 2.9 Hz,1H, Ar), 7.19 (t, $J = 7.2$ Hz, 1H, Ar), 7.23 (s, 1H, Ar), 7.35 (d, $J = 8.0$ Hz, 2H, Ar), 7.43 (t, $J = 7.7$ Hz, 1H, Ar), 7.66 (d, $J = 8.1$ Hz, 2H, Ar), 7.77 (d, $J = 2.6$ Hz, 1H, Ar), 7.98 (d, $J = 7.8$ Hz, 1H, Ar); 14.8 (bs, 1H, OH); ^{13}C NMR (125 MHz, $CDCl_3$): $\delta_C = 21.6$, 24.0, 24.4, 41.5, 50.2, 55.1, 55.5, 64.5, 95.7, 113.5, 115.4, 116.4, 121.4, 122.8, 124.2, 127.9, 128.4(2C), 129.6 (2C), 131.2, 133.3, 134.2, 140.4, 143.9, 154.0, 159.7, 163.7, 174.6; EI-MS: *m/z* (%): 534 (6, M^+), 414 (2), 377 (12), 347 (14), 310 (94), 279 (100); Anal. calcd for $C_{29}H_{30}N_2O_6S$ (534.18): C 65.15, H 5.66, N 5.24 %. Found: C 65.12, H 5.62, N 5.18 %.

N-(2-((4-Hydroxy-2-oxo-2H-chromen-3-yl)(piperidin-1-yl)methyl)-4-methoxyphenyl)-N,4-dimethylbenzenesulfonamide ***(4k)*** White solid, mp: 216–218 ºC, yield: 0.395 g (72 %). IR (KBr) (υ_{max}, cm^{-1}): 2,959, 1,692, 1,456, 1,340, 1,154; ^{1}H-NMR (400 MHz, $CDCl_3$): $\delta_H = 1.70–1.82$ (m, 2H, CH_2), 1.93–2.01 (m, 4H, $2CH_2$), 2.50 (s, 3H, Me), 3.01–3.08 (m, 1H, NCH), 3.30–3.31 (m, 1H, NCH), 3.37 (s, 3H, NMe), 3.45–3.48 (m, 1H, NCH), 3.73 (s, 3H, OMe), 3.86–3.92 (m, 1H, NCH), 5.97 (s, 1H, NCH), 6.46 (d, $J = 8.9$ Hz, 1H, Ar), 6.69 (dd, $J = 8.9$, 3.0 Hz, 1H, Ar), 7.23 (m, 1H, Ar), 7.28 (s, 1H, Ar), 7.38 (d, $J = 8.0$ Hz, 2H, Ar); 7.44 (m, 1H, Ar); 7.68 (d, $J = 3.0$ Hz, 1H, Ar), 7.70 (d, $J = 8.2$ Hz, 2H, Ar), 8.02 (dd, $J = 7.9$, 1.6 Hz, 1H, Ar), 14.27 (bs, 1H, OH); ^{13}C NMR (125 MHz, $CDCl_3$): $\delta_C = 21.6$, 22.5, 24.9, 25.0, 41.5, 49.8, 54.1, 55.5, 65.3, 95.2, 114.2, 115.7, 116.4, 121.4, 122.8, 124.3, 128.1, 128.4 (2C), 129.6 (2C), 131.2, 133.4, 134.4, 138.6, 144.0, 154.0, 159.6, 164.2, 174.8; EI-MS: *m/z* (%): 548 (8, M^+), 310 (100), 279 (78), 236 (16), 188 (86). Anal. calcd for $C_{30}H_{32}N_2O_6S$ (548.20): C 65.68, H 5.88, N 5.11 %. Found: C 65.34, H 6.01, N 4.71 %.

tert-butyl 4-((2-(N,4-dimethylphenylsulfonamido)-5-methoxyphenyl)(4-hydroxy-2-oxo-2H-chromen-3-yl)methyl)piperazine-1-carboxylate ***(4l)*** White solid, mp: 230–232 ºC, yield: 0.442 g (68 %). IR (KBr) (υ_{max}, cm^{-1}): 3,135, 1,683,



Scheme 1 **a** Synthesis of 2-(alkylamino)benzaldehyde **2a–c**; **b**, **c** synthesis of sulfonamide–aldehydes **3a–e**. Reagents and conditions: **a** MeI or EtI, 1,4-dioxane, reflux,1 h; **b** KOH, H_2O/CH_2Cl_2 (1:1); **c** H_2O_2 (35 %), 48 h; **d** pyridine, dry CH_2Cl_2, styrene sulfonyl chloride, 48 h; **e** pyridine, dry CH_2Cl_2, 4-toluenesulfonyl chloride, 48 h



Scheme 2 Synthesis of coumarin-based sulfonamides **4a–n**

1,453, 1,333, 1,150; ^{1}H-NMR (400 MHz, $CDCl_3$): $\delta_H = 1.40$ (s, 9H, CMe_3), 2.36 (s, 3H, Me), 2.44 (s, 3H, NMe), 3.39 (t, J = 5.2 Hz, 4H, $2NCH_2$), 3.62 (s, 3H, OMe), 3.70 (t, J = 5.7 Hz, 4H, $2NCH_2$), 6.04 (d, J = 8.6 Hz, 1H, Ar), 6.40 (dd, J = 8.6, 3.0 Hz, 1H, Ar), 6.56 (s, 1H, CHN), 7.08 (m, 1H, Ar); 7.19 (s, 1H, Ar), 7.20 (d, J = 8.3 Hz, 2H, Ar), 7.34 (*m,* 1H, Ar), 7.42 (d, J = 8.2 Hz, 2H, Ar), 7.89 (dd, J = 8.0, 1.6 Hz, 1H, Ar), 8.08 (dd, J = 7.9, 1.6 Hz, 1H, Ar), 8.55 (bs, 1H, OH); ^{13}C NMR (125 MHz, $CDCl_3$): δ_C = 21.6, 28.3 (3C), 35.1 (2C), 38.8, 44.6 (2C), 55.2, 81.1, 100.8, 107.2, 111.5, 115.8, 115.9, 120.7, 123.3, 124.9, 127.2, 128.4 (2C), 129.5 (2C), 130.8, 131.3, 133.3, 143.7, 144.0, 154.0, 159.2, 166.9, 169.7, 172.0; EI-MS: *m/z* (%): 649 (7, M^+), 463 (6), 308 (17), 279 (58), 236 (10), 188 (100). Anal. calcd for $C_{34}H_{39}N_3O_8S$ (649.25): C 62.85, H 6.05, N 6.47 %. Found: C 63.13, H 5.98, N 6.68 %.

*N-Ethyl-N-(2-((4-hydroxy-2-oxo-2H-chromen-3-yl) (piperidin-1-yl)methyl)phenyl)-4-methylbenzenesulfonamide (**4m**)* White solid, mp: 188–190 ºC, yield: 0.378 g (71 %). IR (KBr) (υ_{max}, cm^{-1}): 3,034, 1,683, 1,454, 1,337, 1,155; ^{1}H-NMR (400 MHz, $CDCl_3$): δ_H = 0.84 (t, J = 7.1 Hz, 3H, NCH_2CH_3), 1.68–1.77 (m, 2H, CH_2), 1.80–1.89 (m, 4H, $2CH_2$), 2.41 (s, 3H, Me), 2.95–3.04 (m, 2H, NCH_2), 3.09–3.15 (m, 1H, NCH), 3.76–4.15 (m, 3H, NCH, NCH_2), 5.82 (s, 1H, NCH), 6.75 (dd, J = 8.0, 1.3 Hz, 1H, Ar), 7.11 (d, J = 8.1 Hz, 1H, Ar), 7.14–7.19 (m, 2H, Ar), 7.26 (m, 1H, Ar), 7.30 (d, J = 8.1 Hz, 2H, Ar), 7.37 (m, 1H, Ar), 7.71 (d, J = 8.2 Hz, 2H, Ar), 7.94 (dd, J = 7.9, 1.7 Hz, 1H, Ar), 8.13 (d, J = 7.9 Hz, 1H, Ar), 13.96 (bs, 1H,OH); ^{13}C NMR (125 MHz, $CDCl_3$): δ_C = 12.5, 21.6, 22.5, 24.9, 24.9, 47.5, 49.8, 53.9, 65.4, 94.5, 116.4, 122.9, 124.4, 127.9, 128.2, 129.3 (2C), 129.4, 129.8 (2C), 130.2, 131.0, 131.3, 136.7, 137.8, 138.6, 144.0, 154.0, 164.4, 175.3; EI-MS: *m/z* (%): 532 (4, M^+), 198 (18), 169 (5), 75 (100), 63 (47), 50 (78); Anal. calcd for $C_{30}H_{32}N_2O_5S$ (532.20): C 67.65, H 6.06, N 5.26 %. Found: C 67.37, H 6.32, N 5.48 %.

*tert-Butyl 4-((2-(N-ethyl-4-methylphenylsulfonamido) phenyl)(4-hydroxy-2-oxo-2H-chromen-3-yl)methyl)piperazine-1-carboxylate (**4n**)* White solid, mp: 223–225 ºC, yield: 0.418 g (66 %). IR (KBr) (υ_{max}, cm^{-1}): 2,983, 1,692, 1,452, 1,343, 1,155; ^{1}H-NMR (400 MHz, $CDCl_3$): δ_H = 0.50 (t, J = 7.3 Hz, 3H, NCH_2CH_3), 1.40 (s, 9H, CMe_3); 2.10 (s, 3H, Me), 2.94–3.15(m, 2H, NCH_2), 3.39 (t, J = 5.1 Hz, 4H, $2NCH_2$), 3.70 (t, J = 5.3 Hz, 4H, $2NCH_2$), 6.30 (dd, J = 7.8, 1.4 Hz, 1H, Ar), 6.71 (s, 1H, NCH), 6.94 (m, 1H, Ar), 7.08 (m, 1H, Ar), 7.19 (d, J = 11.3 Hz, 2H, Ar), 7.33 (m, 1H, Ar), 7.41 (m, 1H, Ar), 7.44 (*d*, J = 8.2 Hz, 2H, Ar), 7.65 (d, J = 7.8 Hz, 1H, Ar), 7.87 (dd, J = 8.0, 1.7 Hz, 1H, Ar), 8.09 (dd, J = 8.1, 1.6 Hz, 1H, Ar), 8.67 (s, 1H, OH); ^{13}C NMR (125 MHz, $CDCl_3$): δ_C = 12.5, 21.6, 28.3 (3C), 34.7, 44.5 (2C), 46.7 (2C), 81.1, 101.8, 107.1, 115.7, 120.0, 123.5, 125.0, 126.3, 128.0, 128.6, 128.6, 129.4, 130.8, 131.0, 131.3, 134.8, 142.8, 143.9, 152.7, 166.7, 170.5, 171.9; EI-MS: *m/z* (%): 633 (3, M^+), 604 (1), 587 (1), 91 (58), 57 (100); Anal. calcd for $C_{34}H_{39}N_3O_7S$ (633.25): C 63.96, H 6.02, N 6.78 %. Found: C 64.13, H 6.25, N 6.82 %.

Results and discussion

Initially, *N*-methyl-2-aminobenzaldehydes **2a–c** (Scheme 1a) and sulphonamide–aldehydes **3a–b** were prepared on the basis of the previously reported method starting from quinolines **1a–c** (Scheme 1b) in three steps [32] and (*E*)-2-phenylethenesulfonyl chloride, respectively [33]. Sulphonamide–aldehydes **3c–e** were then prepared from condensation of aldehydes **2a–c** and *p*-Toluenesulfonyl chloride (Scheme 1c) in the presence of pyridine in CH_2Cl_2 within 48 h in moderate to good yields. Sulfonamide–aldehydes **3c–e** were novel and were completely characterized using their analytical and spectral data. For example, the ^{1}H NMR spectrum of **3c** exhibited characteristic singlets at δ 2.45, 3.21 and 3.86 for Me, NMe and OMe together with a singlet at 10.41 due to CHO. The ^{13}C NMR of **3c** showed

Table 1 Isolated yields obtained for **4a–n**

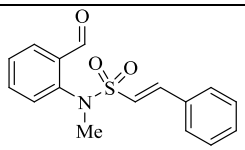
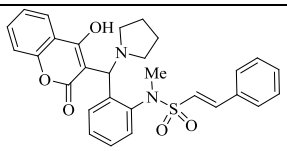
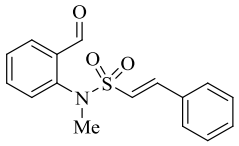
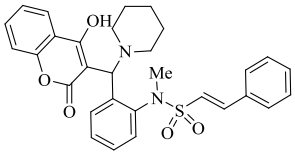
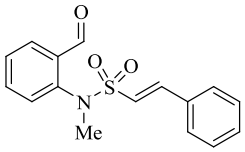
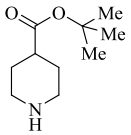
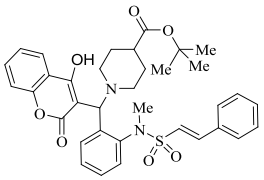
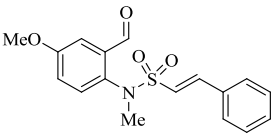
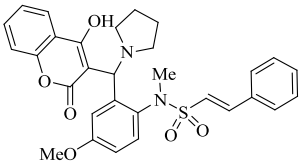
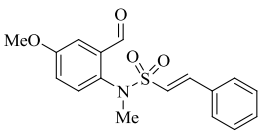
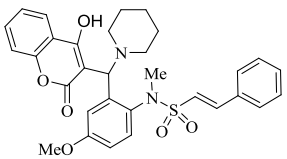
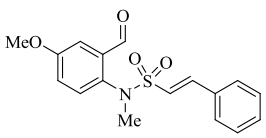
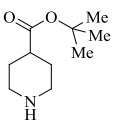
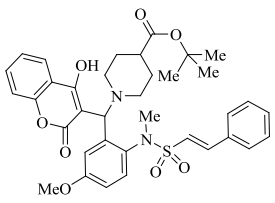
Entry	Sulfonamide aldehyde	Cyclic amine	Product	Yield (%)
1	 **3a**		 **4a**	71
2	 **3a**		 **4b**	65
3	 **3a**		 **4c**	59
4	 **3b**		 **4d**	69
5	 **3b**		 **4e**	60
6	 **3b**		 **4f**	56

Table 1 continued

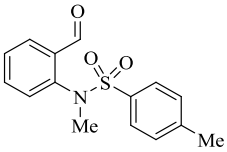
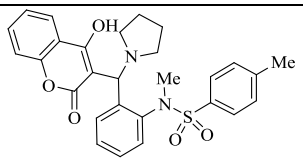
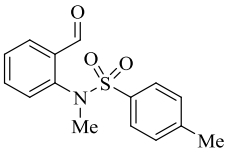
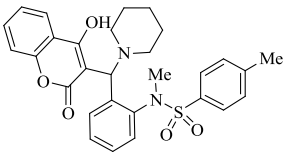
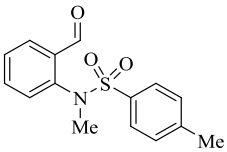
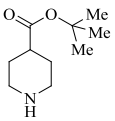
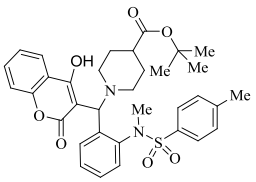
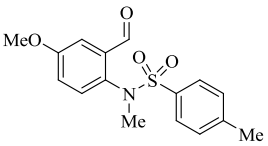
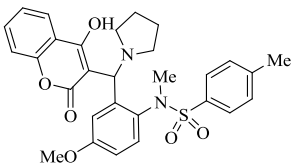
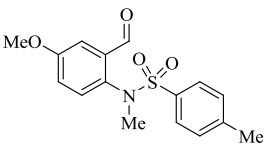
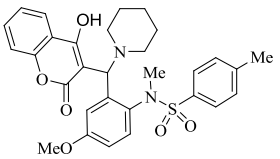
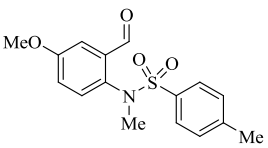
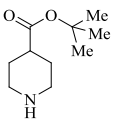
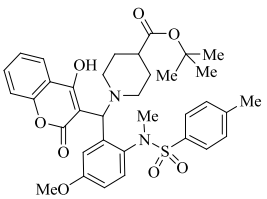
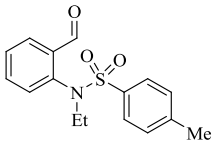
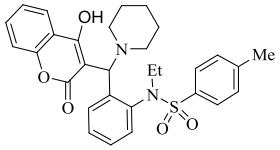
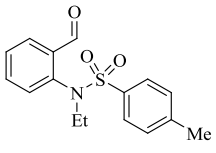
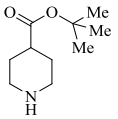
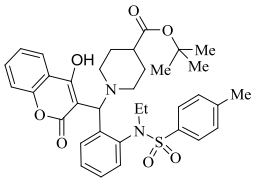
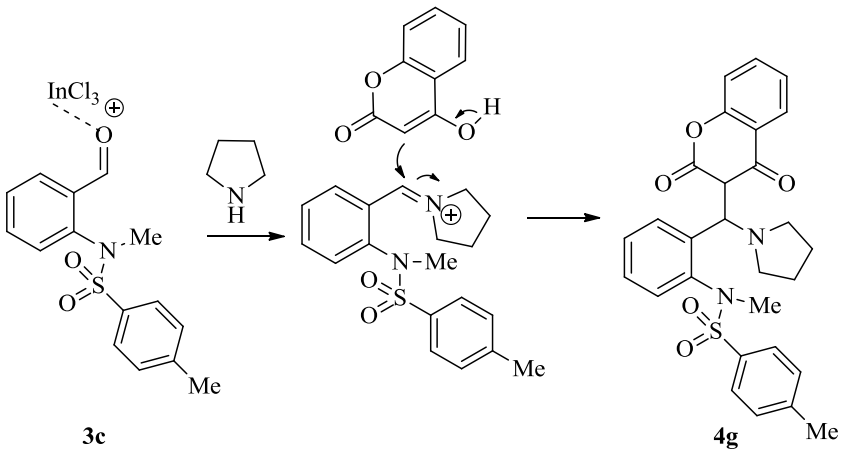
Entry	Sulfonamide-aldehyde	Cyclic amine	Product	Yield (%)
7	 **3c**		 **4g**	79
8	 **3c**		 **4h**	74
9	 **3c**		 **4i**	69
10	 **3d**		 **4j**	80
11	 **3d**		 **4k**	72
12	 **3d**		 **4l**	68

Table 1 continued

Entry	Sulfonamide-aldehyde	Cyclic amine	Product	Yield (%)
13	 **3e**		 **4m**	71
14	 **3e**		 **4n**	66

Scheme 3 A plausible reaction mechanism for the formation of **4g**



14 distinct signals including one appearing at δ 189.9 for CO.

In a model experiment, reaction of sulfonamide–aldehyde **3a** with 4-hydroxycoumarin and pyrrolidine in CH_2Cl_2 using the protocol reported recently [34] failed to show any progress based on TLC results. Gratifyingly, condensation of **3a–e** with 4-hydroxycoumarin and a number of secondary cyclic amines in the presence of 10 % $InCl_3$ in toluene using the Rao and co-workers' method [35] afforded **4a–n** in moderate to good yields within 3 h at ambient temperature (Scheme 2; Table 1). The structure of compounds **4a–n** was confirmed by their analytical and spectral data. For example, the mass spectra of **4d** displayed the molecular ion peak at 516 consistent with the molecular structure. The ^{1}H NMR spectrum of **4d** exhibited characteristic signals at δ 3.32 (s, 3H, MeN), 3.70 (s, 3H, MeO), 5.96 (s, 1H, CHN), 6.88 (d, 1H, J = 15.5 Hz, $CHSO_2$), 7.48 (d, 1H, J = 15.5 Hz, $CH{=}CSO_2$) and 14.42 (bs, 1H, OH) in agreement with the molecular structure. The ^{13}C NMR of **4d** showed 28 distinct signals including 7 peaks appearing at δ 23.9–95.5 due to sp^3 carbons, 21 peaks displaying at δ95.5–174.8 for sp^2 carbons consistent with the proposed structure.

As illustrated in Table 1, cyclic secondary amines such as pyrrolidine, piperidine and piperazine-*N*-Boc are tolerated in these one-pot three-component reactions. Notably, whereas no pronounced reduction in yields is observed in unsubstituted or substituted sulfonamide–aldehydes (compare entries 1–3 with 4–6 and 7–9 with 10–12, Table 1), a regular decrease of 5–8 % in product yields is observed by replacement of pyrrolidine with piperidine or piperazine-*N*-Boc (compare entries 1–3, 4–6, 7–9 and 10–12, Table 1). Such effect seems to be due the bulkier effect of the latter two six-membered secondary amines. On the other hand,

compared to (*E*)-2-phenylethenesulfonyl chloride which affords **4a–f**, utilization of 4-toluenesulfonyl chloride as sulfonating reagent results in the corresponding sulfonamide–aldehydes **4g–n** in higher yields (compare entries 1–6 with 7–12, Table 1). Either lower reactivity of (*E*)-2-phenylethenesulfonyl chloride or partial participation in competing reactions [36] may be responsible for the formation of **4a–f** in lower yields. Whether or not this proposal confirmed is a subject which is currently under investigation in our laboratory.

As Rao et al. [35] have pointed out, the reaction is presumed to have proceeded via the formation of minimum intermediate from secondary cyclic amine with aldehyde. Subsequent nucleophilic addition of 4-hydroxy coumarin results in the formation of product (Scheme 3).

Conclusion

In conclusion, a number of coumarin-based sulphonamides were synthesized via condensation of *N*-(2-formylphenyl)-*N*-methylbenzenesulfonamides **3a–e**, 4-hydroxycoumarin and cyclic secondary amines in toluene. An important aspect of our syntheses which deserves some considerations is the one-pot procedure, easy separation of products by filtration, low reaction time and doing reactions under mild conditions. These new structures broaden the chromenone scaffolds and many of them may represent interesting pharmacophores.

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