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MECHANOCHEMICAL RAPID SYNTHESIS OF NOVEL THIAZOLES LINKED TO 2*H*-CHROMEN-2-ONE MOIETY

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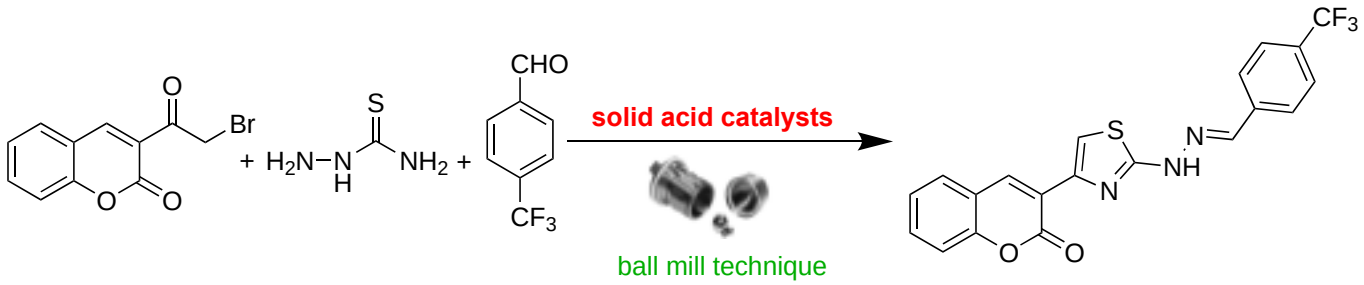
Abstract – A green mechanochemistry synthesis protocol achieved a one-pot synthesis of novel thiazoles linked to 2*H*-chromen-2-one. Chemical transformations were performed in a ball mill without requiring bulk dissolution of reactants. Using a simple one-pot reaction, the compounds were synthesized with good yields and in a short reaction time. Under ball-milling conditions, thiosemicarbazide, 3-(bromoacetyl)-2*H*-chromen-2-one, and aromatic aldehyde were milled in a three-component one-pot reaction utilizing silica triflate (STF) as solid acid catalyst. The reactions were completed in 20 min in 90-98% yields. The catalyst STF show good sustainability in which reused for six runs. ^{1}H NMR, ^{13}C NMR, and mass spectroscopy were used to characterize the synthesized compounds.

Green chemistry has the primary inspiration for the rediscovery of mechanochemistry in organic synthesis.[1] Chemical transformations are achieved by milling or grinding without bulk dissolution of reactants, which is the hallmark of mechanochemistry.[2] Several parameters associated with ball milling can be optimized for reactivity, including the frequency and medium-to-sample weight ratio, that are enclosed in solvent-free reaction environments.[3,4] Among five-membered heterocyclic compounds, 1,3-thiazole is an important structural analog with an extensive range of biological activities.[5-8] On the other hand, there is also many reports proving that 2*H*-chromen-2-one derivatives possess selective anti-cancer effects on different cancer cell lines.[9-12] In view of our interest in developing green synthetic pathways for the synthesis of heterocyclic compounds for biological screening purposes,[13-18] the present study was aimed to synthesize new 2*H*-chromen-2-one linked to thiazole analogues *via* a green mechanochemistry synthesis protocol that avoids

some reported drawbacks released from different reaction pathways that used for the synthesis of 2*H*-chromen-2-one/thiazole such as limited substrate scope, use of organic solvents, low yields, lengthy reaction times and complicated workup procedures that require column chromatographic purification.[19-22] Initially, we optimized the one-pot three-component reaction of equimolar amounts of 3-(2-bromoacetyl)-2*H*-chromen-2-one (**1a**), thiosemicarbazide, and 4-trifluromethylbenzaldehyde (**2a**), in the presence of different solid acid catalysts utilizing ball mill technique to generate 3-(2-(2-(4-(trifluoromethyl)benzylidene)hydrazinyl)thiazol-4-yl)-2*H*-chromen-2-one (**3a**) (Scheme 1).



Scheme 1. Optimizing the reaction conditions for the synthesis of 2*H*-chromen-2-one/thiazole analog

The first step involves studying the above model reaction over different solid acid catalysts such as silica triflate, silica sulfuric acid, acidic alumina, and montmorillonite K10 (Table 1). These catalysts are chosen based on their low environmental impact and reusability.

Table 1. Catalyst screening for the synthesis of **3a** under ball mill technique

Entry	Catalyst	Time	Yield
1	silica triflate (STF)	15 min	99%
2	silica sulfuric acid (SSA)	30 min	92%
3	acidic alumina	60 min	75%
4	montmorillonite K10	30 min	94%

Table 1 shows that the best yield attained 99% of the desired product **3a** using silica triflate (STF) in only 15 min. (entry1). Furthermore, a relatively lower product's yield in longer period of time (94%, 30 min and 92%, 30 min) was attained using montmorillonite K10 and silica sulfuric acid (SSA) catalysts, respectively (entries 4,2). Moreover, when using acidic alumina as a catalyst, the obtained yield 75% in 60 min. (entry 3).

To get the best-optimized reaction conditions for the best solid catalyst (STF), we performed a run of experiments under different parameters such as milling frequency and impact force (controlled in ball milling by varying the number and size of milling balls). The reaction progress was monitored using thin-layer chromatography (TLC) for the model reaction (Scheme 1) under ball milling. The results obtained from the catalytic test reaction were cited in Table 2.

Table 2. Optimizing the reaction conditions* for the synthesis of **3a** under different ball mill conditions using STF catalyst

Entry	Frequency	STF Catalyst weight (g)	Number of milling balls**	Reaction time	Yield
1	30 Hz	0.4	1	30 min	87%
2	**30 Hz**	**0.4**	**2**	**15 min**	**99%**
3	30 Hz	0.4	3	15 min	97%
4	30 Hz	0.5	2	15 min	99%
5	30 Hz	0.3	2	15 min	93%
6	15 Hz	0.4	2	120 min	60%
7	20 Hz	0.4	2	90 min	77%
8	25 Hz	0.4	2	45 min	83%

*Reaction conditions: Equimolar amounts of 3-(2-bromoacetyl)-2*H*-chromen-2-one (**1a**), thiosemicarbazide, and 4-trifluromethylbenzaldehyde (**2a**), in presence of 0.4 g STF catalyst.

**Ball diameter 12 mm.

According to Table 2, the best time was 15 min, as determined by TLC when using two balls of 12 mm diameter and carrying out the reaction at 30 Hz (entry 2). For product **3a**, a minimum of two milling balls are required for an accurate quantitative yield (entries 1-3). Also, the best reaction time and yield attained at frequency 30 Hz with catalyst amount 0.4 g (entry 2, 4, 5). Additionally, 15 Hz, 20 Hz, and 25 Hz (entries 6-8) exhibit longer reaction times and lower yields.

The spectroscopic data of the obtained product is in complete agreement with the structure 3-(2-(2-(4-(trifluoromethyl)benzylidene)hydrazinyl)thiazol-4-yl)-2*H*-chromen-2-one (**3a**). The ^{1}H NMR of compound **3a** shows that three singlet signals at δ 7.82, 8.14 and 8.56 due to ethylenic proton, chromenone-H4 and thiazole proton respectively, also, signals due to aromatic protons in addition to the D_2O exchangeable signal at δ 12.5 due to NH protons (*cf. Supporting Information).* Noteworthy, sustainability of STF catalyst was tested for several reaction runs (taking the synthesis of **3a** as example under the optimized reaction conditions). As soon as the reaction was complete, the catalyst was removed by filtration

(*cf.* Experimental part), then placed in hot ethyl acetate and ultrasonically cleaned to remove any adsorbed organic materials, and then dried. Using the same reaction conditions, the recovered catalyst was reused six times. Regenerated catalyst continues to perform well under the same reaction conditions after six usages, as shown in Figure 1. During the working up in each step, the catalyst lost weight which may explain the slight decay in its catalytic activity observed on the sixth time.

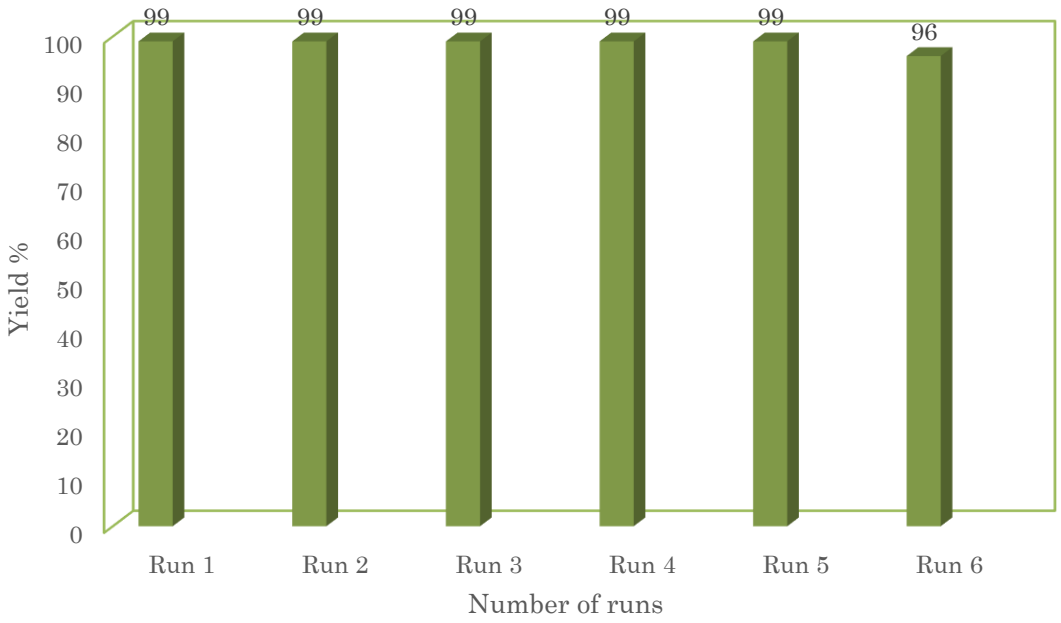
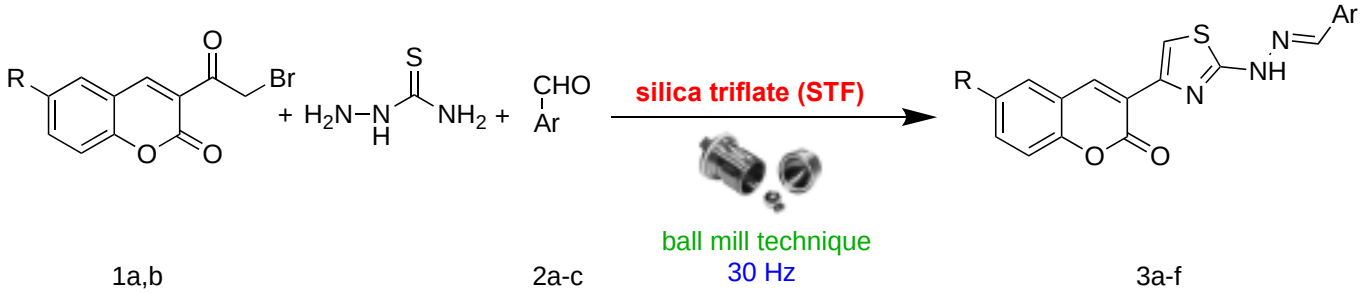


Figure 1. Reusability of STF catalyst

According to Scheme 2, a series of 2*H*-chromen-2-one/thiazole derivatives illustrates the scope and generality of the reaction depicted in Scheme 1.



Scheme 2. Synthesis of the 2*H*-chromen-2-one/thiazole derivatives under optimized conditions

The products **3a-f** were obtained in excellent yields (96-99%) (Figure 2). All the obtained structures in Figure 2 give satisfied elemental analysis and spectroscopic data (*cf.* Experimental section).

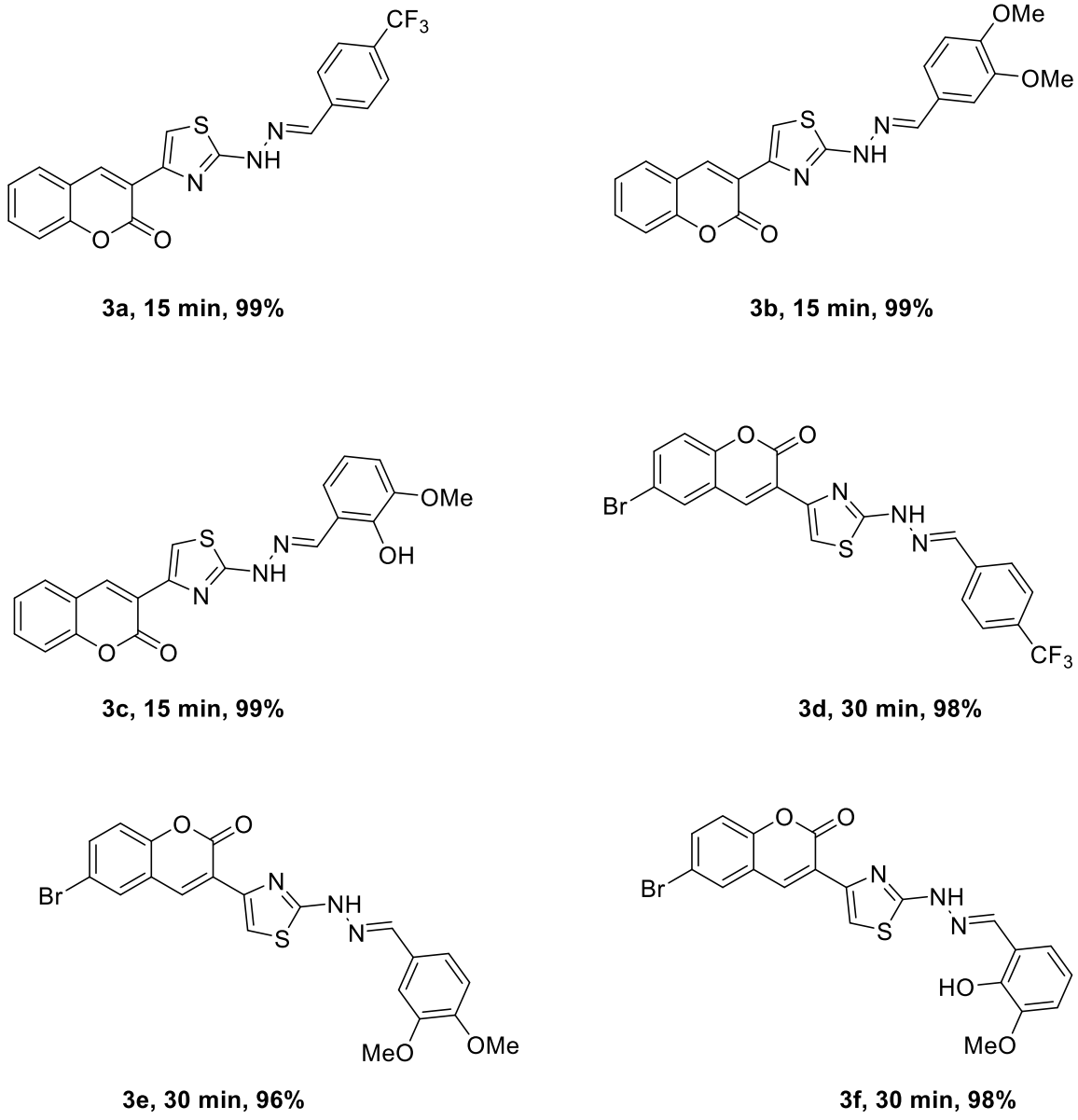


Figure 2. Structures of the synthesized 2*H*-chromen-2-one/thiazole derivatives

In conclusion, a facile and efficient green method has been developed for the synthesis of 2*H*-chromen-2-one/thiazole derivatives catalyzed by STF utilizing ball mill technique. As a result of this protocol, mild reaction conditions are maintained, and the product is easily isolated in excellent yields.

EXPERIMENTAL

All organic solvents were purchased from commercial sources and used as received unless otherwise stated. All other chemicals were purchased from Merck (Darmstadt, Germany), Sigma-Aldrich (St. Louis, MO) or Acros Organics (part of Thermo Fisher Scientific, Waltham, MA) and used without further purification. Thin-layer chromatography was performed on precoated Merck 60 GF254 silica gel plates with a fluorescent indicator, and ultraviolet irradiation at 254 and 360 nm was used for detection. The melting

points were measured on a Stuart melting point apparatus and not corrected. IR spectra were recorded on a Smart iTR, which is an ultrahigh-performance, versatile attenuated total reflectance sampling accessory on the Nicolet iS10 FT-IR spectrometer (Thermo Fisher Scientific). NMR spectra were recorded at 298 K on a Bruker Avance III 400 (9.4 T, 400.13 MHz for ^{1}H, 100.62 MHz for ^{13}C) spectrometer (Bruker, Billerica, MA) with a 5 mm BBFO probe. Chemical shifts (δ in ppm) are relative to internal standard DMSO-d_6 (δ 2.50) for ^{1}H NMR. Mass spectra were recorded on a Thermo Fisher Scientific ISQ single-quadrupole GC–MS. Elemental analyses were carried out on a Euro Vector C, H, N, S analyzer (EA3000 series). Single-crystal X-ray diffraction analysis was performed using a Bruker APEX2. The ball mill was a Retch MM2000 (Hanna, Germany). Silica triflate,[23] silica sulfuric acid,[24] and 3-(2-bromoacetyl)-2*H*-chromen-2-one derivatives **1a,b**[25] were prepared according to the reported literature respectively.

General procedure for the synthesis of 2*H*-chromen-2-one/thiazole derivatives (3a-f)

STF catalyst (0.4 g) was added to a mixture of 3-(2-bromoacetyl)-2*H*-chromen-2-one (**1a**) (10 mmol), thiosemicarbazide (10 mmol), and 4-trifluromethylbenzaldehyde (**2a**) (10 mmol) in a mortar, the grinded blend, utilizing a pestle at ambient temperature, inserted in the 50 mL stainless steel jar equipped with two stainless steel balls (12 mm in diameter). The jar was locked, and milling was carried out at the frequency illustrated in Table 2. After 5 min, thin layer chromatography (TLC) was applied to follow up the reaction progress. If necessary, the milling cycle was repeated until the reaction was complete. The product was isolated by simple washing of the crude reaction residue with hot EtOAc and sonicated for 5 min to desorb all adsorbed product on the surface of the catalyst, then remove the catalyst by filtration. Then the filtrate was concentrated in vacuo under reduced pressure and the residual solid was taken in EtOH and filtered as a pure product. Washing of the pre-used catalyst with EtOAc and drying prior to new catalytic test was systematically followed. Compound **3a** was attained utilizing different catalysts (*cf.* Table 1) using ball mill technique. All the tested catalysts showed reasonable efficiency, but STF provided excellent effectiveness with a high yield in a short period (Table 1). The synthesized compounds' representative spectral and analytical data were depicted in the Supporting Information file.

Spectroscopic and analytical data of compounds 3a-f

3-(2-(2-(4-(Trifluoromethyl)benzylidene)hydrazinyl)thiazol-4-yl)-2*H*-chromen-2-one(3a): mp > 300 ^{o}C; IR (KBr) ν/cm^{-1}: 3302 (NH), 1727 (C=O), 1591 (C=N). ^{1}H NMR (400 MHz, DMSO-d_6), δ: 7.41 (t, J = 7.5 Hz 1H, ArH), 7.47 (d, J = 8.5 Hz, 1H, Ar-H), 7.62-7.66 (m, 1H, ArH), 7.78-7.81 (m, 2H, Ar-H), 7.82 (s, 1H, N=CH), 7.85-7.88 (m, 3H, ArH), 8.14 (s, 1H, chromenone-H4), 8.56 (s, 1H, thiazole proton), 12.48 (s, 1H, NH, D_2O exchangeable); ^{13}C NMR (100 MHz, DMSO-d_6), δ: 111.55, 116.38, 119.63, 121.22, 125.21, 126.20, 127.27, 129.33, 129.90, 130.01, 132.25, 138.76, 140.37, 144.91, 146.12, 152.81, 159.22, 167.96; MS, m/z(%):415 (M^{+}, 89) ; Anal. Calcd for $C_{20}H_{12}F_3N_3O_2S$: C, 57.83; H, 2.91; N, 10.12; S, 7.72. Found: C: 58.02; H, 2.87; N, 10.04; S, 7.66%.

3-(2-(2-(3,4-Dimethoxybenzylidene)hydrazinyl)thiazol-4-yl)-2*H*-chromen-2-one (3b): mp > 300 °C; IR (KBr) *v*/cm^{-1}: 3310 (NH), 1727 (C=O), 1601 (C=N). ^{1}H NMR (400 MHz, DMSO-*d*$_6$), δ: 3.80 (s, 3H, OCH_3), 3.82 (s, 3H, OCH_3), 7.02 (d, *J* = 8.4 Hz 1H, ArH), 7.19 (d, *J* = 8.4 Hz, 1H, Ar-H), 7.28-7.29 (m, 1H, ArH), 7.38-7.47(m, 2H, Ar-H), 7.62-7.66 (m, 1H, ArH), 7.76 (s, 1H, N=CH), 7.86 (d, *J* = 6.4 Hz, 1H, ArH), 8.01(s, 1H, chromenone-H4), 8.55 (s, 1H, thiazole proton), 12.08 (s, 1H, NH, D_2O exchangeable); ^{13}C NMR (100 MHz, DMSO-*d*$_6$), δ: 55.86, 56.03, 108.76, 110.37, 113.22, 116.37, 119.21, 121.04, 125.21, 127.35, 129.28, 130.12, 133.23, 142.47, 143.11, 145.65, 149.51, 150.68, 152.78, 160.11, 168.24; MS, *m/z*(%): 407 (M^+, 91) ; Anal. Calcd for $C_{21}H_{17}N_3O_4S$: C, 61.91; H, 4.21; N, 10.31; S, 7.87. Found: C: 62.09; H, 4.18; N, 10.23; S, 7.80%.

3-(2-(2-(2-Hydroxy-3-methoxybenzylidene)hydrazinyl)thiazol-4-yl)-2*H*-chromen-2-one (3c): mp > 300 °C; IR (KBr) *v*/cm^{-1}: 3412 (OH), 3299 (NH), 1726 (C=O), 1603 (C=N). ^{1}H NMR (400 MHz, DMSO-*d*$_6$), δ: 3.83 (s, 3H, OCH_3), 6.84 (t, *J* = 7.2 Hz, 1H, ArH), 6.98 (d, *J* = 7.4 Hz, 1H, Ar-H), 7.26 (d, *J* = 7.4 Hz, 1H, Ar-H), 7.40 (t, *J* = 7.8 Hz, 1H, Ar-H), 7.48 (d, *J* = 7.6 Hz, 1H, Ar-H), 7.64 (t, *J* = 7.4 Hz, 1H, Ar-H), 7.77 (s, 1H, N=CH), 7.86 (d, *J* = 6.9 Hz, 1H, Ar-H), 8.39(s, 1H, chromenone-H4), 8.55 (s, 1H, thiazole proton), 9.43 (s, 1H, OH, D_2O exchangeable), 12.16 (s, 1H, NH, D_2O exchangeable); ^{13}C NMR (100 MHz, DMSO-*d*$_6$), δ: 56.34, 110.91, 113.07, 116.36, 119.65, 119.75, 121.03, 122.25, 125.21, 128.26, 129.31, 132.19, 138.69, 140.90, 145.92, 148.48, 152.51, 152.79, 159.49, 167.96 ; MS, *m/z*(%): 393 ($M^{+,}$ 89) ; Anal. Calcd for $C_{20}H_{15}N_3O_4S$: C, 61.06; H, 3.84; N, 10.68; S, 8.15. Found: C: 61.25; H, 3.79; N, 10.59; S, 8.10%.

6-Bromo-3-(2-(2-(4-(trifluoromethyl)benzylidene)hydrazinyl)thiazol-4-yl)-2*H*-chromen-2-one (3d): mp > 300 °C; IR (KBr) *v*/cm^{-1}: 3316 (NH), 1723 (C=O), 1599 (C=N). ^{1}H NMR (400 MHz, DMSO-*d*$_6$), δ: 7.43 (d, *J* = 8.8 Hz 1H, ArH), 7.76-7.81 (m, 3H, Ar-H), 7.85-7.88 (m, 2H, ArH), 7.89 (s, 1H, N=CH), 8.14 (s, 1H, chromenone-H5), 8.15 (s, 1H, chromenone-H4), 8.46 (s, 1H, thiazole proton), 12.49 (s, 1H, NH, D_2O exchangeable); ^{13}C NMR (100 MHz, DMSO-*d*$_6$), δ: 112.42, 116.85, 118.63, 121.63, 126.25, 127.29, 130.11, 131.20, 133.75, 134.44, 137.32, 140.48, 143.29, 144.29, 151.81, 159.56, 167.19; MS, *m/z*(%): 492 (M^+, 63); Anal. Calcd for $C_{20}H_{11}BrF_3N_3O_2S$: C, 48.60; H, 2.24; N, 8.50; S, 6.49. Found: C: 48.77; H, 2.22; N, 8.41; S, 6.43%.

6-Bromo-3-(2-(2-(3,4-dimethoxybenzylidene)hydrazinyl)thiazol-4-yl)-2*H*-chromen-2-one (3e): mp > 300 °C; IR (KBr) *v*/cm^{-1}: 3308 (NH), 1726 (C=O), 1601 (C=N). ^{1}H NMR (400 MHz, DMSO-*d*$_6$), δ: 3.85 (s, 3H, OCH_3), 3.87 (s, 3H, OCH_3), 7.06 (d, *J* = 8 Hz 1H, ArH), 7.22 (d, *J* = 8 Hz, 1H, Ar-H), 7.33 (s, 1H, ArH), 7.48 (d, *J* = 8.2 Hz, 1H, Ar-H), 7.77-7.84 (m, 2H, ArH), 8.06 (s, 1H, N=CH), 8.18 (s, 1H, chromenone-H5), 8.19 (s, 1H, chromenone-H4), 8.53 (s, 1H, thiazole proton), 12.16 (s, 1H, NH, D_2O exchangeable); ^{13}C NMR (100 MHz, DMSO-*d*$_6$), δ: 55.85, 56.05, 108.75, 111.73, 112.18, 116.83, 118.61, 120.91, 124.22, 127.51, 131.14, 137.15, 142.57, 143.95, 145.06, 150.69, 151.78, 158.78, 167.02; MS,

m/z(%): 485 (M+, 79); Anal. Calcd for $C_{21}H_{16}BrN_3O_4S$: C, 51.86; H, 3.32; N, 8.64; S, 6.59. Found: C: 52.04; H, 3.28; N, 8.56; S, 6.53%.

6-Bromo-3-(2-(2-(2-hydroxy-3-methoxybenzylidene)hydrazinyl)thiazol-4-yl)-2*H*-chromen-2-one (3f): mp > 300 °C; IR (KBr) *v*/cm^{-1}: 3432 (OH), 3308 (NH), 1721 (C=O), 1603 (C=N). ^{1}H NMR (400 MHz, DMSO-d_6), δ: 3.83 (s, 3H, OCH_3), 6.84 (t, *J* = 7.9 Hz, 1H, ArH), 6.98 (d, *J* = 6.7 Hz, 1H, Ar-H), 7.26 (d, *J* = 6.7 Hz, 1H, Ar-H), 7.43 (d, *J* = 8.8 Hz, 1H, Ar-H), 7.76 (d, *J* = 8.8 Hz, 1H, Ar-H), 7.81 (s, 1H, N=CH), 7.87-7.88 (m, 1H, ArH), 8.15 (s, 1H, chromenone-H5), 8.39(s, 1H, chromenone-H4), 8.49 (s, 1H, thiazole proton), 9.42 (s, 1H, OH, D_2O exchangeable), 12.18 (s, 1H, NH, D_2O exchangeable); ^{13}C NMR (100 MHz, DMSO-d_6), δ: 56.34, 111.77, 113.08, 116.84, 118.05, 118.61, 119.75, 121.03, 121.66, 131.17, 134.83, 137.24, 140.18, 144.27, 145.94, 148.48, 151.79, 158.78, 168.04; MS, *m/z*(%): 470 (M+, 77) ; Anal. Calcd for $C_{20}H_{14}BrN_3O_4S$: C, 50.86; H, 2.99; N, 8.90; S, 6.79. Found: C: 51.05; H, 2.94; N, 8.83; S, 6.72%.

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