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RESEARCH ARTICLE

Heterocycles derived from dimethyldithioimidocarbonates of thiadiazole and thiazole

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A set of imidazolidine, benzimidazole, benzothiazole and benzoxazole derivatives of 1,3,4-thiadiazoles and thiazoles were prepared from corresponding dimethyldithioimidocarbonates by the reaction with various binucleophiles. The structures of the compounds were elucidated and they were screened for antitubercular activity against *Mycobacterium tuberculosis* $H_{37}Rv$ using the BACTEC 460 radiometric system and antibacterial activity against *E. coli* and *B. cirrhosis*, antifungal activity against *A. niger* and *P. worthmanni.*

Keywords: Dimethyldithioimidocarbonate; Imidazolidines; Antitubercular activity; Antimicrobial activity

1. Introduction

Thiazoles and thiadiazoles are useful heterocycles having pharmacological and biological activities such as antimicrobial [1–3], antituberculosis [4], anti-inflammatory [5], anticonvulsant [6, 7], antihypertensive [8, 9], local anasthetic, anticancer [10, 11] and hypoglycemic activities. In recent years much attention has been devoted to the synthesis of these heterocycles as antitubercular and antimicrobial agents. The treatment of mycobacterial infections especially tuberculosis has become an important problem due to the emergence of multidrug resistance. It is reported that the thiadiazole derivatives showed good activity against *Mycobacterium tuberculosis* [12]. This observation had an impact on the synthesis and search of some derivatives of thiadiazole to study their biological properties.

The compounds **2a–d** have been synthesized by treating corresponding 2-amino-1,3,4-thiadiazole/2-aminothiazole with carbon disulfide in alkaline medium, followed by methylation using methyl iodide. The chemical formula and molecular structure of dimethyldithioimidocarbonates **2a–d** were established by analytical and spectral data. The IR spectrum of **2a–d** showed the absence of $\upsilon_{N\text{-}H}$ and its ^1H NMR spectrum displayed a singlet for six protons around δ 2.5 due to equivalent -SCH_3 groups. Heterocyclization of compounds

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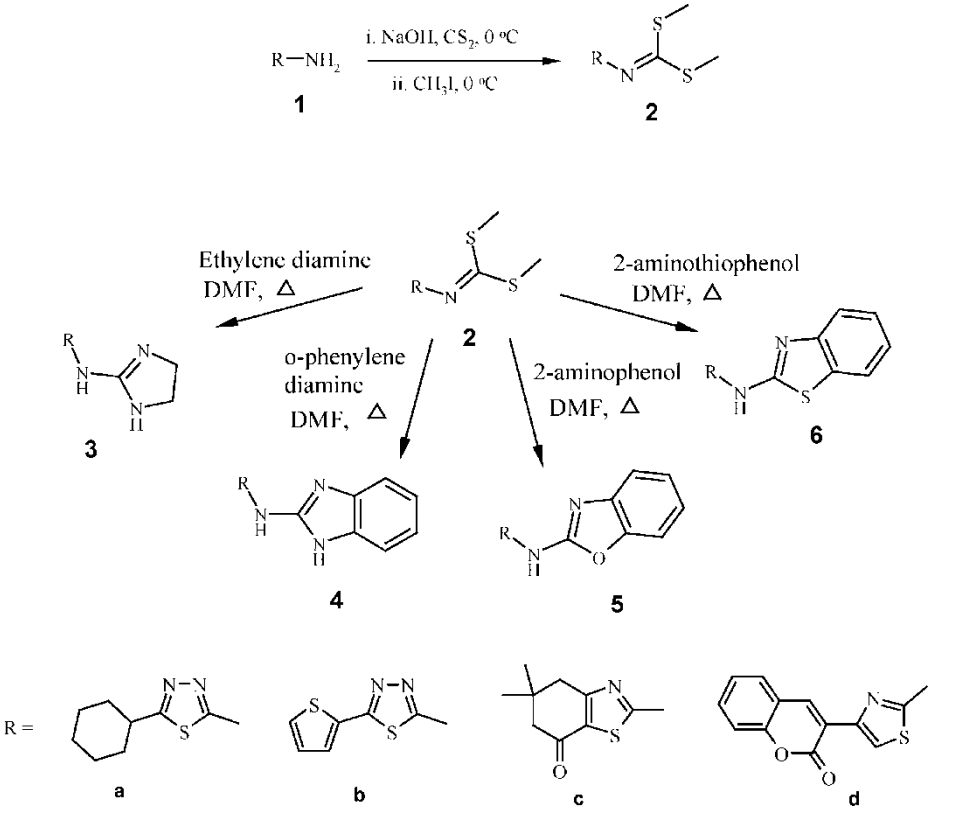
2a–d with various binucleophiles *viz*, ethylenediamine, *o*-phenylenediamine, 2-aminophenol and 2-aminothiophenol in DMF at 120 °C resulted in the formation of imidazolidines **3a–d**, benzimidazoles **4a–d**, benzoxazoles **5a–d** and benzothiazoles **6a–d**, respectively, in high yields. The products **3a–d** obtained by the reaction of **2a–d** with ethylenediamine possessed the strong $\upsilon_{N\text{-}H}$ band around 3330 cm^{-1} in IR and the ^1H NMR displayed a D_2O exchangeable broad singlet in the region δ 7.00–8.00 assigned for both the NH protons and a singlet around δ 3.50 for four protons, confirming the fact that both hydrogens are being equivalent due to the delocalization of proton over imidazolidine and linker nitrogen atoms. Similarly the formation of various other heterocycles *viz*, benzimidazoles **4a–d**, benzoxazoles **5a–d** and **6a–d** was established by their spectral and analytical data. All the products showed the N-H stretching frequencies in the region 3200–3240 cm^{-1} in their IR spectra and the NH protons were observed in the range between δ 7.00 to 12.00 as D_2O exchangeable broad singlet in ^1H NMR (NH protons not observed when the solvent used is $CDCl_3$ + TFA). The compounds were also confirmed by ^{13}C NMR spectra (scheme 1).

All the newly synthesized compounds were tested for their antitubercular activity against *M. tuberculosis* [13, 14], antibacterial activity [15, 16] against *Escherichia coli and Bacillus cirrhosis* and antifungal activity [17] against *Penicillium wortmannii and Aspergillus niger.* Among the compounds tested, **2b**, **3d**, **4b** showed percentage inhibition 28, 10 and 19 for *M. tuberculosis*, respectively. Compounds **2b**, **6d** and **4a**, **5b** showed good antibacterial activity against *E. coli and B. cirrhosis* compared to standard Norfloxacin. However, none of the tested compounds displayed good antifungal activity.



SCHEME 1

2. Experimental

Melting points are uncorrected. TLC was carried out on aluminium silica gel 60 F_{254} (Merck) detected by UV light and iodine vapors. IR spectra were obtained from Nicolet Impact-410 FT-IR spectrophotometer, using KBr pellets. ^{1}H NMR and ^{13}C NMR spectra were recorded on Bruker AC-300F 300 MHz, spectrometer in $CDCl_3$ and DMSO-d_6 using $SiMe_4$ as an internal standard. Mass spectrum was recorded on Micromass Walter spectrometer by electron impact technique and elemental analysis was carried out using Heraus CHN rapid analyzer.

2-Amino-5-cyclohexyl-1,3,4-thiadiazole (**1a**), 2-amino-5-(2-thienyl)-1,3,4-thiadiazole (**1b**), 2-amino-5,5-dimethyl-5,6-dihydro-1,3-benzothiazol-7(*4H*)-one (**1c**) and 2-amino-4(3′-coumarinyl)thiazole (**1d**) were prepared as per the literature methods [18–20].

2.1 *Preparation of dimethyl-5-alkyl/aryl-2-yl-1,3,4-thiadiazol-2-yldithioimidocarbonate (2a–d). General procedure*

To a well stirred cold solution of 2-amino-5-alkyl/aryl-1,3,4-thiadiazole/2-aminothiadiazole (0.01 mol) in dimethylformamide (8 mL), were added sodium hydroxide solution (20M, 4 mL), carbon disulfide (1.52 g, 0.02 mol) and methyl iodide (3.384 g, 0.024 mol) in sequence at an interval of 30 min, stirring was continued for 3 hrs, the mixture was then poured into cold water and the resulting sticky solid was extracted with chloroform, washed with water and dried over anhydrous sodium sulphate. The combined extracts were evaporated to dryness under vacuum and recrystallized from chloroform-hexane mixture to yield the corresponding dimethyldithioimidocarbonate (**2a–d**) in moderate yields as crystalline solids.

2.1.1 Dimethyl-5-cyclohexyl-1,3,4-thiadiazol-2-yldithioimidocarbonate (2a). Yield 56%, colorless solid (chloroform + hexane); mp 48–50 °C; IR (KBr) υ 3070, 2928, 1620 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 1.23–1.90 (m, 10H, cyclohexyl), 2.54 (s, 6H, SCH_3), 3.03 (m, 1H, C1-H, cyclohexyl); Anal. Calcd for $C_{11}H_{17}N_3S_3$: C, 45.96; H, 5.96; N, 14.62; Found: C, 46.44; H, 6.11; N, 14.99%.

2.1.2 Dimethyl-5-thien-2-yl-1,3,4-thiadiazol-2-yldithioimidocarbonate (2b). Yield 56%, pale yellow solid (chloroform + hexane); mp 108–110 °C; IR (KBr) υ 3079, 1530 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 2.62 (s, 6H, SCH_3), 7.09 (dd, $J_{H3H4} = 6$ Hz, $J_{H4H5} = 6$ Hz, 1H, C4-H, thienyl), 7.45 (d, $J = 6$ Hz, 1H, C3-H, thienyl), 7.48 (d, $J = 6$ Hz, 1H, C5-H, thienyl); Anal. Calcd for $C_9H_9N_3S_4$: C, 37.60; H, 3.16; N, 14.62; Found: C, 37.84; H, 3.32; N, 14.78%.

2.1.3 Dimethyl-5,5-dimethyl-7-oxo-4,5,6,7-tetrahydro-1,3-benzothiazol-2-yldithioimidocarbonate (2c). Yield 55%, pale yellow solid (chloroform + hexane); mp 95-97 °C; IR (KBr) υ 1661, 1600 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 1.14 (s, 6H, gemdimethyl), 2.46 (s, 2H, CH_2), 2.62 (s, 6H, SCH_3), 2.86 (s, 2H, CH_2CO); ^{13}CNMR (DMSO-d_6) δ 17.98, 28.97, 35.27, 41.44, 52.40, 118.62, 163.49, 174.29, 192.16 and 197.00; Anal. Calcd for, $C_{12}H_{16}N_2OS_3$: C, 47.97; H, 5.37; N, 9.32. Found: C, 48.34; H, 5.82; N, 9.39%.

2.1.4 Dimethyl-4-(2-oxo-2*H*-chromen-3-yl)-1,3-thiazol-2-yldithioimidocarbonate (2d). Yield 45%, yellow crystalline solid (chloroform + hexane); mp 140–142 °C; IR (KBr) υ 1728, 1606 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 2.64 (s, 6H, SCH_3), 7.28–7.76 (m, 4H, coumarinyl), 8.28 (s, 1H, C5-H, thiazole), 8.64 (s, 1H, C4-H, coumarin); ^{13}C NMR ($CDCl_3$) δ 16.4, 116.6, 118.4,

120.0, 121.3, 124.8, 128.7, 131.6, 139.6, 145.5, 153.3, 160.0, 167.6 and 173.0; Anal. Calcd for $C_{15}H_{12}N_2O_2S_3$: C, 51.70; H, 3.47; N, 8.04; Found: C, 51.86; H, 3.31; N, 8.33%.

2.2 *Preparation of 5-cyclohexyl-N-imidazolidin-2-ylidene-1,3,4-thiadiazol-2-amine (3a–d). General procedure*

A mixture of **2** (0.002 mol) and ethylenediamine (0.14 g, 0.0024 mol) in dimethylformamide (10 mL) was refluxed for 6 hrs. The solid that separated after cooling was collected by filtration, washed with water, alcohol, dried and recrystallized from ethanol. Yield 80%, colorless needles (ethanol); mp 124–126 °C; IR (KBr) υ 3256, 3048, 1639, 1538 cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.24–1.90 (m, 10H, cyclohexyl), 3.11 (m, 1H, C_1H, cyclohexyl), 3.52 (s, 4H, imidazolidine), 7.60 (s, 2H, NH, D_2O exchangeable); Anal. Calcd for $C_{11}H_{17}N_5S$: C, 52.56; H, 6.82; N, 27.86; Found: C, 53.00; H, 6.99; N, 28.33%.

2.2.1 ***N*-Imidazolidin-2-ylidene-5-thien-2-yl-1,3,4-thiadiazol-2-amine (3b).** Yield 80%, pale yellow needles (ethanol); mp 144–146 °C; IR (KBr) υ 3338, 1615 cm^{-1}; ^1H NMR (DMSO-d_6) δ 3.55 (s, 4H, imidazolidine), 7.13 (dd, $J_{H3H4} = 3$ Hz, $J_{H4H5} = 6$ Hz, 1H, C4-H, thienyl), 7.42 (d, $J = 3$ Hz, 1H, C3-H, thienyl), 7.65 (d, $J = 6$ Hz, 1H, C5-H, thienyl), 7.68 (s, 2H, NH, D_2O exchangeable); Anal. Calcd for $C_9H_9N_5S_2$: C, 43.01; H, 3.61; N, 27.86; Found: C, 43.08; H, 3.92; N, 28.38%.

2.2.2 2-(Imidazolidin-2-ylideneamino)-5,5-dimethyl-5,6-dihydro-1,3-benzothiazol-7(4*H*)-one (3c). Yield 71%, pale yellow solid (ethanol); mp 118–120 °C; IR (KBr) υ 3332, 3213, 1640, 1616, 1600 cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.14 (s, 6H, gem dimethyl), 2.42 (s, 2H, CH_2), 2.71 (s, 2H, CH_2CO), 3.77(s, 4H, imidazolidine), 7.83 (br s, 2H, NH, D_2O exchangeable); ^{13}C NMR ($CDCl_3$) δ 28.35, 35.75, 41.22, 43.49, 51.22, 117.18, 124.35, 165.59, 165.98 and 195.11; Anal. Calcd for, $C_{12}H_{16}N_4OS$: C, 54.52; H, 6.10; N, 21.19; Found: C, 54.71; H, 6.24; N, 21.42%.

2.2.3 3-[2-(Imidazolidin-2-ylideneamino)-1,3-thiazol-4-yl]-2*H*-chromen-2-one (3d). Yield 70%, yellow solid (ethanol); mp 234–236 °C; IR (KBr) υ 3339, 1724, 1639 cm^{-1}; ^1H NMR ($CDCl_3$) δ, 4.07 (s, 4H, imidazolidine), 7.44–7.72 (m, 4H, coumarin), 8.04 (s, 1H, C5-H, thiazole), 8.37 (br s, 2H, NH, D_2O exchangeable), 8.47 (s, 1H, C4-H, coumarin); Anal. Calcd for $C_{15}H_{12}N_4O_2S$: C, 57.68; H, 3.87; N, 17.94; Found: C, 57.73; H, 3.96; N, 18.10%.

2.3 *Preparation of 5-cyclohexyl-N-1,3-dihydro-2H-benzimidazol-2-ylidene-1,3,4-thiadiazol-2-amine (4a). General procedure*

A mixture of **2** (0.002 mol) and *o*-phenylenediamine (0.26 g, 0.002 mol) was refluxed in dimethylformamide (10 mL) for 8 hrs. The separated solid was collected by filtration, washed with aqueous alcohol, dried and recrystallized from ethanol. Yield 72%, colorless prisms (ethanol); mp 148–150 °C; IR (KBr) υ 3218, 2929, 2848, 1627, 1601, 1565 cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.26–1.87 (m, 10H, cyclohexyl), 3.07(m, 1H, C1-H, cyclohexyl), 7.30 (m, 2H, C5, C6-H, benzimidazole), 7.39 (m, 2H, C4, C7-H, benzimidazole), 11.08 (s, broad, 2H, NH, D_2O exchangeable); Anal. Calcd for $C_{15}H_{17}N_5S$: C, 60.17; H, 5.72; N, 23.39; Found: C, 60.61; H, 5.81; N, 23.76%.

2.3.1 *N*-1,3-Dihydro-2H-benzimidazol-2-ylidene-5-thien-2-yl-1,3,4-thiadiazol-2-amine (4b). Yield 72%, pale yellow needles (ethanol); mp 172–174 °C; IR (KBr) υ 3251, 1609 cm^{-1}; ^{1}H NMR (DMSO-d_6) δ 7.13 (dd, $J_{H3H4} = 3$ Hz, $J_{H4H5} = 3$ Hz, 2H, C5, C6-H, benzimidazole), 7.17 (d, $J = 3$ Hz, C4-H, thienyl), 7.38 (d, $J = 3$ Hz, 2H, C4, C7-H, benzimidazole), 7.53 (d, $J = 3$ Hz, 1H, C3-H, thienyl), 7.69 (dd, $J = 3$ Hz, 1H, C5-H, thienyl), 12.01 (s, broad, 2H, NH, D_2O exchangeable); Anal. Calcd for $C_{13}H_9N_5S_2$: C, 52.15; H, 3.03; N, 23.39; Found: C, 52.62; H, 3.33; N, 23.88%.

2.3.2 2-(1*H*-Benzimidazol-2-ylamino)-5,5-dimethyl-5,6-dihydro-1,3-benzothiazol-7(4*H*)-one (4c). Yield 60%, Yellow crystalline solid(ethanol + DMF); mp 260–262 °C; IR (KBr) υ 3218, 1656, 1619, 1578 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 1.21 (s, 6H, gemdimethyl), 2.62 (s, 2H, CH_2), 2.98 (s, 2H, CH_2CO), (m, 4H, Ar-H), 11.48 (br, s, 2H, NH, D_2O exchangeable); ^{13}C NMR ($CDCl_3$) δ 28.02, 35.79, 40.75, 50.92, 113.27, 113.39, 117.06, 120.84, 122.88, 126.58, 128.22, 144.90, 160.52, 166.52 and 196.01; Anal. Calcd for $C_{16}H_{16}N_4OS$: C, 61.52; H, 5.16; N, 17.93; Found: C, 61.66; H, 5.07; N, 17.99%.

2.3.3 3-[2-(1*H*-Benzimidazol-2-ylamino)-1,3-thiazol-4-yl]-2H-chromen-2-one (4d). Yield 64%, yellow granules (ethanol + DMF); mp 230–232 °C; IR (KBr) υ 3139, 1720, 1635, 1603 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 7.47–7.81 (m, 8H, Ar-H), 8.02 (s, 1H, C5-H, thiazole), 8.68 (s, 1H, C4-H, coumarin), 11.03 (b s, 2H, NH, D_2O exchangeable); Anal. Calcd for $C_{19}H_{12}N_4O_2S$: C, 63.32; H, 3.36; N, 15.55; Found: C, 63.66; H, 3.54; N, 15.95%.

2.4 *Preparation of N-(5-cyclohexyl-1,3,4-thiadiazol-2-yl)-1,3-benzoxazol-2-amine (5a)*

A solution of **2** (0.002 mol) and 2-aminophenol (0.26 g, 0.0024 mol) in dimethylformamide (12 mL) was refluxed for 8 hrs. The separated solid was collected by filtration, washed with aqueous alcohol, dried and recrystallized. Yield 69%, yellow solid (methanol + DMF); mp 220–222 °C; IR (KBr) υ 3331, 2899, 1634, 1598 cm^{-1}; ^{1}H NMR ($CDCl_3$ + TFA) δ 1.28–1.91 (m, 10H, cyclohexyl), 3.14 (m, 1H, C1-H, cyclohexyl), 7.24–7.70 (m, 4H, Ar-H); Anal. Calcd for $C_{15}H_{16}N_4OS$: C, 59.98; H, 5.37; N, 18.65; Found: C, 60.13; H, 5.46; N, 18.90%.

2.4.1 *N*-(5-Thien-2-yl-1,3,4-thiadiazol-2-yl)-1,3-benzoxazol-2-amine (5b). Yield 55%, red crystalline solid (methanol + DMF); mp 225–227 °C; IR (KBr) υ 3300, 1598 cm^{-1}; ^{1}H NMR ($CDCl_3$ + TFA) δ 7.23–7.67(m, 7H, Ar-H); ^{13}C NMR ($CDCl_3$ + TFA) δ 111.7, 113.3, 113.6, 126.4, 127.9, 128.4, 129.9, 131.2, 132.2, 146.3, 155.3 and 157.3; Anal. Calcd for $C_{13}H_8N_4OS_2$: C, 51.98; H, 2.68; N, 18.65; Found: C, 52.38; H, 2.79; N, 18.84%.

2.4.2 2-(1,3-Benzoxazol-2-ylamino)-5,5-dimethyl-5,6-dihydro-1,3-benzothiazol-7(4*H*)-one (5c). A mixture of **2** (0.002 mol) and 2-aminophenol (0.26 g, 0.0024 mol) was refluxed in dimethylformamide (12 mL) for 8 hrs, the completion of the reaction was confirmed by TLC. The solid that separated after cooling was collected by filtration, washed with ethanol, dried and recrystallized. Yield 55%, yellow crystalline solid (DMF); mp 274–276 °C; IR (KBr) υ 3288, 1654, 1598 cm^{-1}; ^{1}H NMR ($CDCl_3$ + TFA) δ 1.23 (s, 6H, gemdimethyl), 2.67 (s, 2H, CH_2), 2.91 (s, 2H, CH_2CO), 7.49–7.66 (m, 4H, Ar-H), 9.05 (br, s, 1H, NH, D_2O exchangeable) Anal. Calcd for, $C_{16}H_{15}N_3O_2S$: C, 61.32, H, 4.82; N, 13.41; Found: C, 61.09; H, 4.69; N, 13.78%.

2.4.3 3-[2-(1,3-Benzoxazol-2-ylamino)-1,3-thiazol-4-yl]-2*H*-chromen-2-one (5d). Yield 65%, Yellow solid (ethanol + DMF); mp 248–250 °C; IR (KBr) υ 3369, 1725, 1590 cm^{-1}; ^1H NMR ($CDCl_3$ + TFA) δ 7.41–7.79 (m, 8H, Ar-H), 7.96 (s, 1H, C_5-H, thiazole), 8.52(s, 1H, C4-H, coumarin); Anal. Calcd for $C_{19}H_{11}N_3O_3S$: C, 63.15; H, 3.07; N, 11.63; Found: C, 63.66; H, 3.39; N, 11.81%.

2.5 *Preparation of N-(5-Cyclohexyl-1,3,4-thiadiazol-2-yl)-1,3-benzothiazol-2-amine (6a)*

A solution of **2** (0.002 mol) and 2-aminothiophenol (0.3 g, 0.0024 mol) in dimethylformamide (12 mL) was refluxed for 8 hrs (monitored by TLC). The solid separated was collected by filtration, washed with aqueous ethanol, dried and recrystallized. Yield 65%, orange amorphous solid (methanol + DMF); mp 234–236 °C; IR (KBr) υ 3314, 2891, 1629, 1598 cm^{-1}; ^1H NMR ($CDCl_3$ + TFA) δ 1.26–1.89 (m, 10H, cyclohexyl), 3.12 (m, 1H, C1-H, cyclohexyl), 7.22–7.67 (m, 4H, Ar-H); Anal. Calcd for $C_{15}H_{16}N_4S_2$: C, 56.93; H, 5.10; N, 17.71; Found: C, 56.98; H, 5.38; N, 17.90%.

2.5.1 *N*-(5-Thien-2-yl-1,3,4-thiadiazol-2-yl)-1,3-benzothiazol-2-amine (6b). Yield 58%, yellow solid (methanol); mp 220–222 °C; IR (KBr) υ 3220, 1609 cm^{-1}; ^1H NMR ($CDCl_3$ + TFA) δ 7.24–7.69 (m, 7H, Ar-H); Anal. Calcd for $C_{13}H_8N_4S_3$: C, 49.34; H, 2.55; N, 17.71; Found: C, 49.21; H, 2.86; N, 17.73%.

2.5.2 2-(1,3-Benzothiazol-2-ylamino)-5,5-dimethyl-5,6-dihydro-1,3-benzothiazol-7(4*H*)-one (6c). Yield 48%, yellow granules (ethanol + DMF); mp 252–254 °C; IR (KBr) υ 3328, 1654, 1599 cm^{-1}; ^1H NMR ($CDCl_3$ + TFA) δ 1.26 (s, 6H, gem dimethyl), 2.71 (s, 2H, CH_2) 2.95 (s, 2H, CH_2CO), 7.57–7.93 (m, 4H, Ar-H); ^{13}C NMR($CDCl_3$ + TFA) δ 27.9, 35.7, 37.2, 50.6, 112.9, 115.8, 116.7, 123.3, 125.7, 127.0, 130.1, 135.9, 154.8, 168.3 and 193.9; Anal. Calcd for $C_{16}H_{15}N_3OS_2$: C, 58.33; H, 4.59; N, 12.75; Found: C, 58.81; H, 4.46; N, 12.95%.

2.5.3 3-[2-(1,3-Benzothiazol-2-ylamino)-1,3-thiazol-4-yl]-2*H*-chromen-2-one (6d). Yield 61%, intense yellow solid (ethanol + DMF); mp 220–222 °C; IR (KBr) υ, 3141, 1722, 1605 cm^{-1}; ^1H NMR ($CDCl_3$) δ 7.45–7.91 (m, 8H, Ar-H), 8.20 (s, 1H, C5-H, thiazole), 8.67 (s, 1H, C4-H, coumarin), 11.93 (b s, 1H, NH, D_2O exchangeable); ^{13}C NMR ($CDCl_3$) δ 112.9, 114.5, 115.8, 116.6, 117.1, 117.5, 119.1, 122.9, 125.1, 126.2, 126.5, 129.4, 129.6, 133.8, 136.5, 139.7, 141.9, 153.1 and 165.0; Anal. Calcd for $C_{19}H_{11}N_3O_2S_2$: C, 60.46; H, 2.94; N, 11.13; Found: C, 60.28; H, 3.16; N, 11.37%. Mass, m/z (%): 378 (M + 1, 100%).

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