International Journal of ChemTech Research
CODEN (USA): IJCRGG ISSN : 0974-4290
Vol.4, No.1, pp 149-151, Jan-Mar 2012

Studies in the Series of Condensed Benzothiazole Thiazolidinone Systems with Bridge Nitrogen Atom

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Abstract: 2-Hydrazino-4,6-dimethylbenzothiazole (**2**) has been prepared by refluxing 2-amino-4,6-dimethyl benzothiazole (**1**) in ethanol with hydrazine hydrate. Compound (**2**) was condensed with different aromatic aldehydes to form corresponding hydrazones (3_{a-e}). These hydrazones on reaction with mercapto acetic acid afforded corresponding thiazolidin-4-ones (4_{a-e}). The newly synthesized compounds were evaluated for their antibacterial activity.

Key words: 2-Hydrazino-4,6-dimethylbenzothiazole, thiazolidin-4-ones, antibacterial activity.

Introduction:

In our attempts in preparing condensed heterocyclic systems with bridge nitrogen atom we came across many references of the synthesis of benzothiazole azetidinone but very few on the thiazolidinone condensed system. Hence it was considered worth while to probe into bridged benzothiazolo and thiazolidinone compounds. Benzothiazoles and thiazolidinones[1-4] are independently pharmacologically active compounds[5-6]. To investigate the biologically active some such condensed systems, this work was undertaken.

The starting material 2-hydrazino-4,6-dimethylbenzothiazole was prepared by refluxing 2-amino-4,6-dimethylbenzothiazole with hydrazine hydrochloride in ethylene glycol. The product thus obtained was condensed with different aldehydes to yield hydrazones. The hydrazones thus obtained were treated with mercapto acetic acid in the presence of $ZnCl_2$ and DMF. Corresponding 3-(4,6-dimethylbenzothiazole-2-ylamino)-2-arylthiazolidin-4-ones (4_{a-e}) were obtained in good yields (Scheme 1). IR spectra of these newly synthesized compounds (4_{a-e}) exhibited absorption bands at 1650-1600cm^{-1} due to >C=N & >C=O stretching respectively. ^{1}H-NMR spectra exhibited peaks in the region δ 4-5, δ 2-3 & δ 4-6 due to –NH, CH_2 & -CH protons respectively. The appearance of molecular ion peaks in Mass spectra corresponding to molecular weight confirms the structure of proposed thiazolidinones.

Antibacterial Activity:

The synthesized compounds were tested for their antimicrobial activity by paper disc method against *Xanthomonas, Erwinia* and *E-Coli* (gram–ve) using *ampicillin* as a standard antibacterial compound. The antibacterial screening data of the compounds have been incorporated in Table **1**. The synthesized compounds exhibited zone of inhibition of 16-14-14 mm in diameter where as standard ampicillin exhibited zone of inhibition of 18-16-08 mm in diameter against *Xanthomonas, Erwinia* and *E-Coli* respectively. Amongst the synthesized compounds 4_a, 4_c and 4_d showed higher zone of inhibition against *Xanthomonas, Erwinia* and *E-Coli* respectively. It is worth noting that compounds are very effective especially towards *E-Coli* and comparatively more active even than standards.

Experimental Section:

Melting points were determined in open capillaries and are found uncorrected. IR spectra (KBr discs) were recorded on FTIR- SCHIMADZU 84005 and Thermo Nicolet Nexus 670 Spectrophotometer and absorption was expressed in cm^{-1}. NMR spectra were recorded on Gemini 200 MHz spectrometer with Tetramethyl silane as an internal standard. Chemical shift values were mentioned in δ ppm. Mass spectra were recorded on a FT VG-7070 H Mass spectrophotometer using the EI technique at 70 ev. The progress of all reactions was monitored by TLC on 2x5 cm pre-coated silica gel 60 F254 plates of thickness of 0.25 mm (Merck) and spots were visualized under UV 254-366 nm and iodine chamber. The compounds were analyzed for C, H and N.

General Procedure:

3-(4,6-Dimethyl-benzothiazol-2-yl amino)-2-aryl thiazolidin-4-ones (4_{a-e}).

A mixture of a hydrazone, (0.001 mole) mercapto acetic acid in dimethyl formamide, with pinch of fused zinc chloride was refluxed for 5-7 hours on oil bath which was then cooled. The contents were poured over crushed ice. The solid thus separated was treated with saturated sodium bicarbonate solution to remove excess of mercapto acetic acid. The product thus separated was filtered and recrystalized from methanol.

3-(4,6-Dimethyl-benzothiazol-2-ylamino)-2-phenyl-thiazolidin-4-one (4_a): Yield 0.216g, (60%), m.p: above 300 ^oC; ir (KBr): 1581(C=N stretch), 1271 (C-N stretch), 1606 (>C=O stretch). ^1H nmr ($CDCl_3$): 2.1(s, 6H, Ar-CH_3), 3.2(s, 2H, CH_2), 4.3(s, 1H, NH), 5.8(s, 1H, CH), 7.0-7.6(m, 7H, Ar-H). ms: m/z 355(M^+). *Anal*. Calcd. for $C_{18}H_{17}N_3OS_2$: C, 60.84; H, 4.78; N, 11.83. Found: C, 60.32, H, 4.57, N, 11.33.

3-(4,6-Dimethyl-benzothiazol-2-ylamino)-2-(2-hydroxy-3-methoxy-phenyl)-thiazolidin-4-one (4_b): Yield 0.510g, (65%), m.p: 220 ^oC; ir (KBr): 1540 (C=N stretch), 1268 (C-N stretch), 1608 (>C=O stretch). ^1H nmr ($CDCl_3$): 2.3(s, 6H, Ar-CH_3), 2.9(s, 2H, -CH_2), 3.5(s, 3H, OCH_3), 4.0(s, 1H, OH), 4.8(s, 1H, NH), 6.0(s, 1H, CH), 7.2-7.9(m, 5H, Ar-H). ms: m/z 401(M^+). *Anal*. Calcd. for $C_{19}H_{19}N_3O_3S_2$: C, 56.85; H, 4.73; N, 10.47. Found: C, 56.38, H, 4.57, N, 10.21.

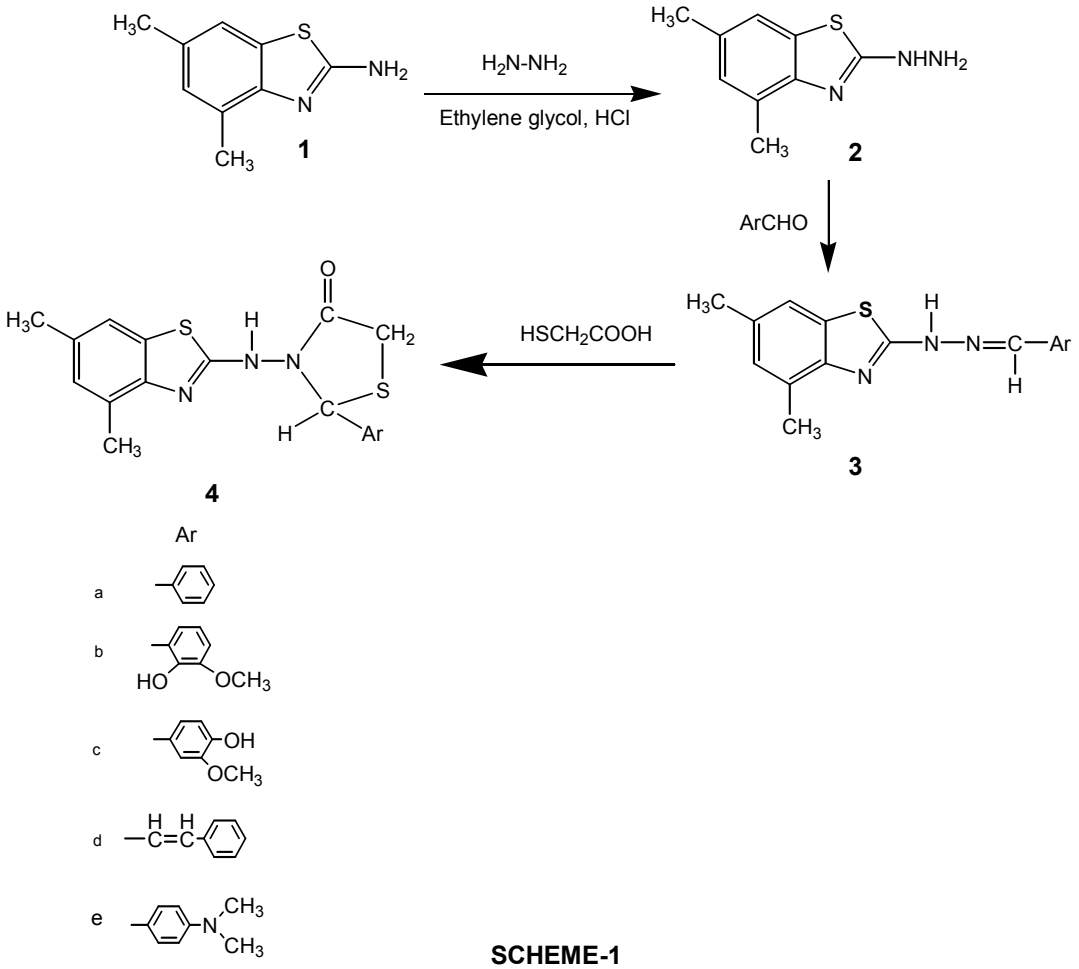
3-(4,6-Dimethyl-benzothiazol-2-ylamino)-2-(4-hydroxy-3-methoxy-phenyl)-thiazolidin-4-one (4_c): Yield 0.553g, (70%), m.p: above 300 ^oC; ir (KBr): 1574 (C=N stretch), 1257(C-N stretch), 1623 (>C=O stretch). ^1H nmr ($CDCl_3$): 2.0(s, 6H, Ar-CH_3), 2.5(s, 2H, CH_2), 3.4(s, 3H, OCH_3), 4.1(s, 1H, OH), 5.0(s, 1H, NH), 5.9(s, 1H, CH), 7.1-7.8(m, 5H, Ar-H). ms: m/z 401(M^+). *Anal*. Calcd. for $C_{19}H_{19}N_3O_3S_2$; C, 56.85; H, 4.73; N, 10.47. Found: C, 56.37; H, 4.19; N, 10.40.

3-(4,6-Dimethyl-benzothiazol-2-ylamino)-2-styryl-thiazolidin-4-one (4_d): Yield 0.522g, (69%), m.p: 140 ^oC; ir (KBr): 1540 (C=N stretch), 1261 (C-N stretch), 1647 (>C=O stretch).^1H nmr ($CDCl_3$): 2.3(s, 3H, Ar-CH_3), 2.6(s, 2H, CH_2), 4.1-4.6(d, 2H, CH=CH), 5.1(s, 1H, NH), 7.2-7.9(m, 7H, Ar-H). ms: m/z 381(M^+). *Anal*. Calcd. for $C_{20}H_{19}N_3OS_2$: C, 62.99; H, 4.98; N, 11.02. Found: C, 62.39; H, 4.53; N, 10.89.

3-(4,6-Dimethyl-benzothiazol-2-ylamino)-2-(4-dimethylamino-phenyl)-thiazolidin-4-one (4_e): Yield 0.558g, (70%), m.p: 150 ^oC; ir (KBr): 1523 (C=N stretch), 1269 (C-N stretch), 1610 (>C=O stretch). ^1H nmr ($CDCl_3$): 2.38 (s, 3H, Ar-CH_3), 2.50 (s, 6H, N-CH_3), 3.02 (s, 2H, -CH_2), 5.0(s, 1H, NH), 5.7 (s, 1H, -CH), 6.6-7.5 (Ar-H). ms: m/z 398(M^+). *Anal*. Calcd. for $C_{20}H_{22}N_4OS_2$: C, 60.30; H, 5.52; N, 14.07. Found: C, 60.11; H, 5.07; N, 14.00.

Acknowledgement

The authors are thankful to Dr.G.M.Kalamse, Principal, Science College, Nanded for laboratory facilities and Dr.K.R.Gangakhedkar, Principal, Pratibha Niketan Mahavidyalaya, Nanded for encouragement.



SCHEME-1

Table 1

Compound	Diameter in mm of zone of inhibition at 25 µg/disc		
	Xanthomonas	Erwinia	E-Coli
4_a	16	12	07
4_b	12	NA	11
4_c	13	14	07
4_d	16	10	14
4_e	12	09	11
Ampicillin (1mg/ml)	18	16	08

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