Asian Journal of Chemistry; Vol. 35, No. 7 (2023), 1575-1578

ASIAN JOURNAL OF CHEMISTRY

https://doi.org/10.14233/ajchem.2023.27601

Synthesis of (*E*)-2-Benzylidene-*N*-(3-(3-oxo-2,3-dihydro-4*H*-benzo[*b*][1,4]oxazin-4-yl)propyl)hydrazine-1-carbothioamides

PALLA SAI LAKSHMI and PULIGILLA SHANKARAIAH*

Department of Pharmacy, Chaitanya Deemed to be University, Hanamakonda-506001, India

*Corresponding author: E-mail: drspuligilla@gmail.com

Received: 2 February 2023; *Accepted*: 12 April 2023; *Published online*: 6 July 2023; AJC-21276

A series of novel substituted-(*E*)-2-benzylidene-*N*-(3-(3-oxo-2,3-dihydro-4*H*-benzo[*b*][1,4]oxazin-4-yl)propyl)hydrazine-1-carbothioamides (**9a-j**) was synthesized in satisfactory to excellent yield by reacting (*Z*)-*N*′-benzylidene hydrazine carbothioamides (**8a-j**) with 4-(3-bromopropyl)-2*H*-benzo[*b*][1,4]oxazin-3(4*H*)-one (**5**) in K_2CO_3 and dry *N*,*N*-dimethyl formamide. The novel carbothioamides (**9a-j**) structures were confirmed using ^{1}H NMR, IR and mass spectroscopic methods.

Keywords: Synthesis, 2*H*-Benzo[*b*][1,4]oxazin-3(4*H*)-one, Substituted carbothioamides.

INTRODUCTION

The strong chemical and biological capabilities of heterocyclic compounds provide a variety of interesting structural issues for medicinal chemists. Researches toward the development of a new heterocyclic ring structure that contains considerable biological activity is ongoing, despite the fact that millions of heterocyclic molecules have already been developed [1-3]. Approximately 99% both natural and synthesized heterocycles have pharmacological activities [4]. Thus, physiologically and pharmacologically active heterocyclic molecules have recently come to the forefront of medical studies.

Numerous pharmacological compounds with intriguing therapeutic potential have been characterized as containing heterocyclic rings with two or three heteroatoms in their skeleton. Moreover, fused different moities of heterocyclic rings are also known to be nitric oxide synthase (NOS) inhibitors, which may be helpful in the treatment of neurological illnesses and inflamatory arthritis [5,6]. Xue *et al*. [7] reported the synthesis of 3,5-dimethyl isoxazole related to benzoxazinones, which are found to be selective bromo-domain and extra-terminal protein inhibitors. By using click chemistry, Rajitha *et al*. [8] synthesized 1,2,3-triazole derivatives of 1,4-benzoxazinones and tested them for their ability to inhibit the proliferation of cervical, pancreatic, breast and neuroblastoma cell lines. Nucleophilic substitution of *N*-alkylated 2-aminophenols by bromo aryl-acetates generated from either (*R*)-pantolactone or l-mandelate exhibits dynamic kinetic resolution, Youk *et al*. [9] synthesized enantiomers of *N*-substituted 3-arylated 3,4-dihydro-1,4-benzoxazin-2-ones. Byun *et al*. [10] also reported 1,4- dihydropyridine mediated alkylation and acylation of C-H bonds in benzoxazinones.

Through the cyclization of chalcones utilizing thiosemicarbazide/semicarbazide in acetic acid as solvent, Rana *et al*. [11] synthesized carbothioamide based pyrazoline derivatives. Capan *et al*. [12] reported the microwave-assisted synthesis of novel hydrazine-1-carbothioamides containing unsubstituted benzimidazole skeleton in a basic medium to efficiently convert them to 1, 2 and 4-triazole derivatives. In light of these promising findings, the synthesis of carbothioamides was conducted using benzoxazinone and hydrazine scaffolds, each of which has a different substituents in the heterocyclic ring. The synthetic reaction involves the *N*-alkylation of the primary amine in benzylidene hydrazine carbothioamide, when reacted with halo-alkyl benzoxazinone.

EXPERIMENTAL

Necessary anhydrous conditions for all reactions be done in dried glasswork. Spectrograph obtained through VNMR GEMINI proton frequency 400, 500 or 300 MHz BRUKER Avance analytical instruments, tetramethanesilane $Si(CH_3)_4$ was used as an internal reference. Infrared spectral analysis

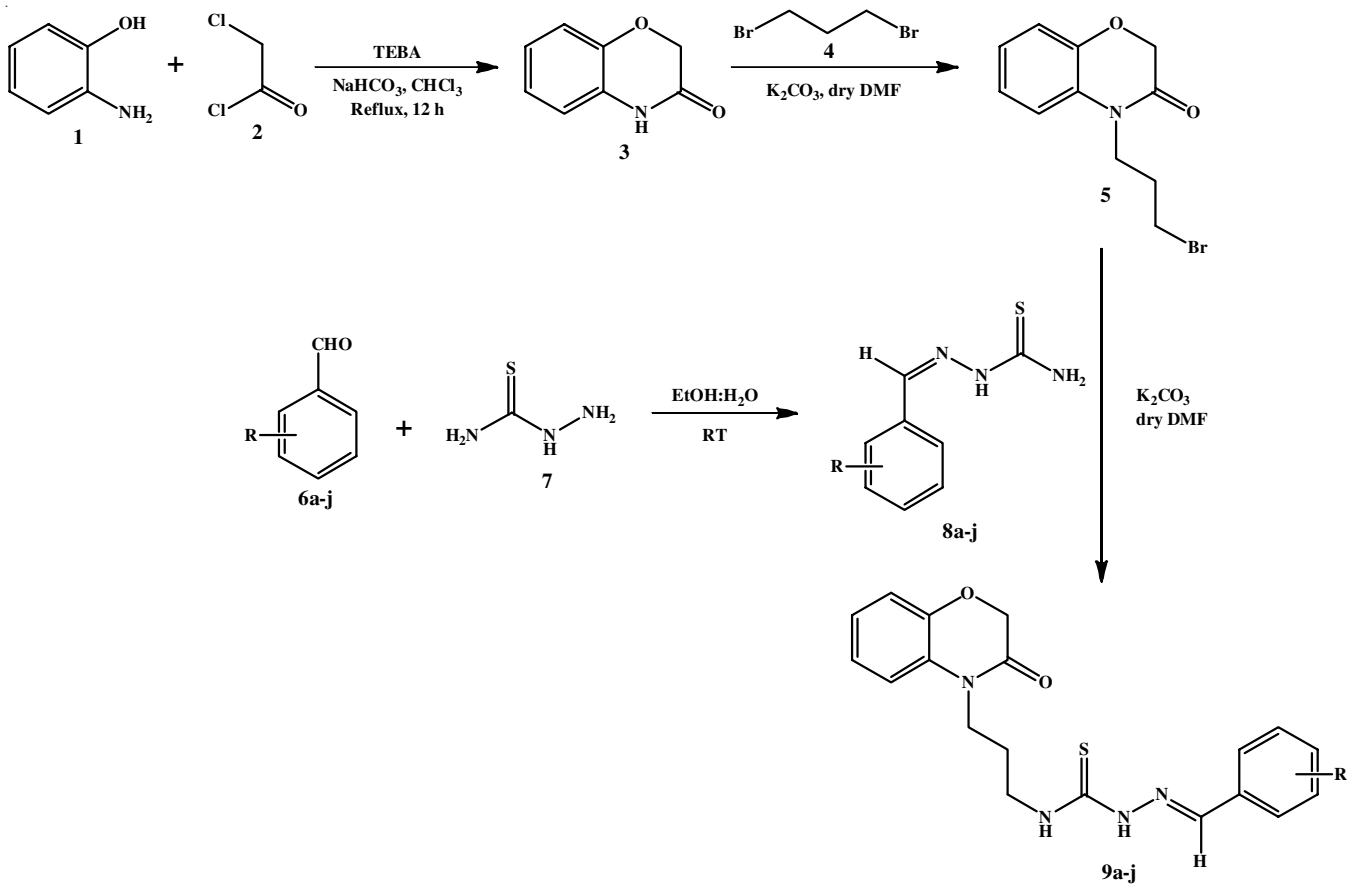
was investigated on Perkin-Elmer 683 or Perten Falling Number® 1310 spectrometers using KBr pellets. The electron ionization MS was obtained on a 70-70 mass spectrometer made by VG Micromass-7070 Hz. A potential of 70 V was applied and electrons were accelerated to 70 eV kinetic energy to generate positive ions.

4-(3-Bromopropyl)-2*H*-benzo[*b*][1,4]oxazin-3(4*H*)-one (5): 2*H*-Benzo[*b*][1,4]oxazin-3(4*H*)- one (**3**, 5 g) was added into a round bottomed flask containing DMF (5 mL) and K_2CO_3 kept in cooling bath at 0 ºC followed by the addition of 1,3-dibromo propane (**4**, 6 g) and stirred for 10 min. Further 12 h were spent at room temperature with the cooling bath reintroduced and stirred. The admixture was poured into a container full of crushed ice and stirred for 10 min. Homogenized extracts should be washed with salt solution (2 × 50 mL) and water (3 × 100 mL) followed by ethyl ethanoate extraction (3 × 100 mL). The organic phase was desiccated in Na_2SO_4 and the product was purified by flash purification chromatography. Yield: 90%, R_f = 1.8, (ethyl acetate/hexane 2:8), b.p.: 191 ºC, brownish liquid. IR (KBr, ν_{max}, cm^{-1}): 3063, 3038, 2915, 1762, 1613, 1487. ^1H NMR ($CDCl_3$) (400 MHz) δ ppm: 2.15-2.25 (multiplet, 2H, -CH_2-), 3.42-3.46 (triplet, 2H, -CH_2-), 4.01-4.07 (triplet, 2H, -N-CH_2-), 4.55 (singlet, 2H, CH_2), 6.90-7.08 (multiplet, 4H, arom.-H). EI-MS calcd. (found) for $C_{11}H_{12}NO_2Br$: 269.01 (270.00).

(*E*)-2-Benzylidene-N-(3-(3-oxo-2,3-dihydro-4*H*-benzo[*b*][1,4]oxazin-4-yl)propyl)hydrazine-1-carbothioamide derivatives (9a-j): 4-(3-Bromopropyl)-2*H*-benzo[*b*][1,4]-oxazin-3(4*H*)-one (50 mg) was added to a round bottom flask containing DMF (5 mL) and K_2CO_3 in an cooling bath at 0 ºC, (*E*)-2-benzylidenehydrazine-1-carbothioamide (**8a**, 60 mg) added and blended for 10 min. The cooling bath put back and whisked about 12 h in room temperature. The admixture be run on crushed ice and agitated for 10 min. After extraction with ethyl ethanoate (3 × 100 mL), the mixed components be cleansed by concentrated salt solution (2 × 50 mL) and H_2O (3 × 100 mL) (**Scheme-I**). The organic phase be desiccated in Na_2SO_4 and crude be clarified by flash purification chromatography to yield the synthesized title compounds.

(*E*)-2-(4-Fluorobenzylidene-*N*-(3-(3-oxo-2,3-dihydro-4*H*-benzo[*b*][1,4]oxazin-4-yl)propyl)hydrazine-1-carbothioamide (9a): R_f = 1.6 (ethyl acetate/hexane 2:8), b.p.: 190 ºC, IR (KBr, ν_{max}, cm^{-1}): 3448, 2927, 1628, 1600, 1386, 1350, 1109. ^1H NMR ($CDCl_3$) (400 MHz): δ 2.20-2.30 (multiplet, 2H, -CH_2-), 3.11-3.20 (triplet, 2H, -CH_2- NH-), 4.00-4.13 (triplet, 2H, -CH_2-NCO-), 4.60 (singlet, 2H, -OCH_2-CO), 5.53 (singlet, 1H, - NH), 6.95-7.00 (multiplet, 4H, aromatic-H), 7.01-7.09 (multiplet, 2H, aromatic-H), 7.62 (doublet, 1H, aromatic-H), 7.80 (doublet, 1H, aromatic-H), 8.28 (singlet, 1H, -CH=N-). ESI-Mass calcd. (found) for $C_{19}H_{19}N_4O_2SF$: 386.12 (387.15).

(*E*)-*N*-(3-(3-Oxo-2,3-dihydro-4*H*-benzo[*b*][1,4]oxazin-4-yl)propyl)-2-(4-(trifluoromethyl)benzylidene)hydrazine-1-carbothioamide (9b): R_f = 1.9 (ethyl acetate/hexane 2:8), b.p.: 187 ºC, IR (KBr, ν_{max}, cm^{-1}): 3423, 3314, 2923, 2853,



Scheme-I: Novel analogues of carbothioamide

1670, 1609, 1412, 1326, 1111, 1060, 746. ^{1}H NMR ($CDCl_3$) (400 MHz): δ 2.10-2.20 (multiplet, 2H, -CH_2-), 3.15-3.20 (triplet, 2H, -CH_2-NH-), 4.10-4.13 (triplet, 2H, -NCH_2), 4.60 (singlet, 2H, -OCH_2- CO), 5.52 (singlet, 1H, -NH), 6.95-7.09 (multiplet, 6H, arom.-H), 7.62 (doublet, 1H, *J* = 8.92 Hz, arom.-H), 7.80 (doublet, 1H, *J* = 8.90 Hz, aromatic-H), 8.28 (singlet, 1H, aromatic -CH=N-). ESI-Mass calcd. (found) for $C_{20}H_{19}N_4O_2SF_3$: 436.12 (437.15).

(*E*)-2-(2-Hydroxybenzylidene)-*N*-(3-(3-oxo-2,3-dihydro-4*H*-benzo[*b*][1,4]oxazin-4-yl)propyl)hydrazine-1-carbothioamide (9c): R_f = 1.8, (ethyl acetate/hexane 2:8), b.p.: 192 °C, IR (KBr, ν_{max}, cm^{-1}): 3441, 3321, 2975, 2927, 1664, 1588, 1527, 1500, 1411, 1274. ^{1}H NMR ($CDCl_3$) (400 MHz): δ 2.02-2.15 (multiplet, 2H, -CH_2-), 3.10-3.20 (triplet, 2H, -CH_2-NH), 4.05-4.13 (triplet, 2H, -CH_2-N-), 4.59 (singlet, 2H, - OCH_2CO), 6.95-7.05 (multiplet, 4H, aromatic-H), 7.10-7.20 (multiplet, 2H, aromatic-H), 7.28 (doublet, 1H, aromatic-H), 8.00 (singlet, 1H, -OH), 8.20 (singlet, 1H, -CH=N-). ESI-Mass calcd. (found) for $C_{19}H_{20}N_4O_3S$: 384.13 (385.15).

(*E*)-2-(4-Hydroxybenzylidene)-*N*-(3-(3-oxo-2,3-dihydro-4*H*-benzo[*b*][1,4]oxazin-4-yl)propyl)hydrazine-1-carbothioamide (9d): R_f = 1.7 (ethyl acetate/hexane 2:8), b.p.: 189 ºC, IR (KBr, ν_{max}, cm^{-1}): 3448, 2964, 2927, 2363, 1628, 1594, 1385, 1351, 1114. ^{1}H NMR ($CDCl_3$) (400 MHz): δ 2.18-2.20 (multiplet, 2H, -CH_2-), 3.12-3.20 (triplet, 2H, -CH_2-NH), 4.10-4.19 (triplet, 2H, -CH_2-N-), 4.60 (singlet, 2H, -OCH_2CO), 6.85 (doublet, 2H, *J* = 8.90 Hz, aromatic-H), 6.95-7.10 (multiplet, 4H, arom.-H), 7.60 (doublet, 2H, *J* = 8.90 Hz, aromatic-H,), 8.20 (singlet, 1H, -CH=N-). ESI-Mass calcd. (found) for $C_{19}H_{20}N_4O_3S$: 384.13 (385.15).

(*E*)-2-Benzylidene-*N*-(3-(3-oxo-2,3-dihydro-4*H*-benzo-[*b*][1,4]oxazin-4-yl)propyl)hydrazine-1-carbothioamide (9e): R_f = 1.9 (ethyl acetate/hexane 2:8), b.p.: 187 ºC, IR (KBr, ν_{max}, cm^{-1}): 3445, 2926, 1625, 1605, 1355, 1100. ^{1}H NMR ($CDCl_3$) (400 MHz): δ 2.01-2.09 (multiplet, 2H, -CH_2-), 2.29-2.39 (triplet, 2H, -CH_2-NH-), 2.57-2.60 (triplet, 2H, -N-CH_2-), 7.05-7.86 (multiplet, 10H, arom.-H), 8.59 (singlet, 1H, -CH=N-). ESI-Mass calcd. (found) for $C_{19}H_{20}N_4O_2S$: 368.13 (369.17).

(*E*)-2-(2-Methoxybenzylidene)-*N*-(3-(3-oxo-2,3-dihydro-4*H*-benzo[*b*][1,4]oxazin-4-yl)propyl)hydrazine-1-carbothioamide (9f): R_f = 1.9 (ethyl acetate/hexane 2:8), b.p.: 189 ºC, IR (KBr, ν_{max}, cm^{-1}): 3422, 3313, 2957, 2923, 1670, 1607, 1524, 1412, 1327, 1112. ^{1}H NMR ($CDCl_3$) (400 MHz): δ 1.87-1.95 (multiplet, 2H, -CH_2-), 3.15-3.20 (triplet, 2H, -CH_2-NH-), 3.87 (singlet, 3H, -OCH_3), 4.02-4.13 (triplet, 2H, -N-CH_2-), 4.60 (singlet, 2H, -OCH_2CO), 4.45 (singlet, 2H, 2×-NH), 6.90-7.08 (multiplet, 5H, arom.-H), 7.20 (doublet, 1H, aromatic-H), 7.35 (triplet, 1H, arom.-H), 7.99 (doublet, 1H, *J* = 8.70 Hz, arom.-H), 8.77 (singlet, 1H, -N=CH-). ESI-Mass calcd. (found) for $C_{20}H_{22}N_4O_3S$: 398.14 (399.15).

(*E*)-2-(3-Methoxybenzylidene)-*N*-(3-(3-oxo-2,3-dihydro-4*H*-benzo[*b*][1,4]oxazin-4-yl)propyl)hydrazine-1-carbothioamide (9g): R_f = 1.8 (ethyl acetate/hexane 2:8), b.p.: 192 ºC, IR (KBr, ν_{max}, cm^{-1}): 3448, 2958, 2927, 2363, 1629, 1601, 1358, 1350, 1271, 1107. ^{1}H NMR ($CDCl_3$) (400 MHz): δ 2.10-2.20 (multiplet, 2H, -CH_2-), 3.15-3.20 (triplet, 2H, -CH_2-NH-), 3.85 (singlet, 3H, -OCH_3), 4.10-4.13 (triplet, 2H, -CH_2-N-), 4.60 (singlet, 2H, -OCH_2CO), 5.50 (broad, singlet, 1H, -NH), 6.90-7.08 (multiplet, 6H, arom.-H), 7.25-7.31 (multiplet, 2H, arom.-H), 8.25 (singlet, 1H, -CH=N-). ESI-Mass calcd. (found) for $C_{20}H_{22}N_4O_3S$: 398.14 (399.15).

(*E*)-2-(4-Methoxybenzylidene)-*N*-(3-(3-oxo-2,3-dihydro-4*H*-benzo[*b*][1,4]oxazin-4-yl)propyl)hydrazine-1-carbothioamide (9h): R_f = 1.8 (ethyl acetate/hexane 2:8), b.p.: 192 ºC, IR (KBr, ν_{max}, cm^{-1}): 3449, 2964, 2924, 2854, 2364, 1627, 1358, 1351, 1114, 1008. ^{1}H NMR ($CDCl_3$) (400 MHz): δ 1.80-1.90 (multiplet, 2H, -CH_2-), 2.60-2.7 (triplet, 2H, -CH_2-NH-), 3.80-3.88 (triplet, 2H, -N-CH_2-), 3.85 (singlet, 3H, -OCH_3), 4.60 (singlet, 2H, -OCH_2CO), 6.21 (singlet, 1H, -NH), 6.92-7.50 (multiplet, 4H, Ar-H), 7.20 (doublet, 2H, *J* = 8.82 Hz, arom.-H), 7.30 (doublet, 2H, *J* = 8.82 Hz, arom.-H), 8.00 (singlet, 1H, -CH=N). ESI-Mass calcd. (found) for $C_{20}H_{22}N_4O_3S$: 398.14 (399.15).

(*E*)-2-(4-Cyanobenzylidene)-*N*-(3-(3-oxo-2,3-dihydro-4*H*-benzo[*b*][1,4]oxazin-4-yl-propyl)hydrazine-1-carbothioamide (9i): R_f = 1.7 (ethyl acetate/hexane 2:8), b.p.: 187 ºC, IR (KBr, ν_{max}, cm^{-1}): 3420, 3311, 2957, 2924, 2855, 2222, 1668, 1606, 1509, 1460, 1420, 1225, 1123, 1052, 1003. ^{1}H NMR ($CDCl_3$) (400 MHz): δ 2.10-2.20 (multiplet, 2H, -CH_2-), 3.15-3.20 (triplet, 2H, -CH_2-NH-), 4.10-4.18 (triplet, 2H, -N-CH_2), 4.59 (singlet, 2H, -OCH_2-CO), 5.55 (singlet, 1H, -NH), 6.95-7.10 (multiplet, 4H, aromatic-H), 7.65-7.67 (doublet, 2H, *J* = 8.78 Hz, aromatic-H), 7.80-7.83 (doublet, 2H, *J* = 8.82 Hz, aromatic-H), 8.25 (singlet, 1H, -CH=N). ESI-Mass calcd. (found) for $C_{20}H_{19}N_5O_2S$: 393.13 (394.15).

(*E*)-2-(2-Nitrobenzylidene)-*N*-(3-(3-oxo-2,3-dihydro-4*H*-benzo[*b*][1,4]oxazin-4-yl)propyl)hydrazine-1-carbothioamide (9j): R_f = 1.4 (ethyl acetate/hexane 2:8), b.p.: 192 ºC, IR (KBr, ν_{max}, cm^{-1}): 3446, 2969, 2924, 1643, 1603, 1385, 1351, 1216, 1116. ^{1}H NMR ($CDCl_3$) (400 MHz): δ 2.10-2.20 (multiplet, 2H, -CH_2-), 3.15-3.20 (triplet, 2H, -CH_2-NH-), 4.10-4.18 (triplet, 2H, -N-CH_2), 4.60 (singlet, 2H, -OCH_2CO), 5.50 (singlet, 1H, -NH), 6.95-7.09 (multiplet, 4H, aromatic-H), 7.50-7.58 (triplet, 1H, aromatic-H), 7.60- 7.65 (triplet, 1H, arom.-H), 7.90-7.94 (doublet, 1H, arom.-H), 8.05-8.07 (doublet, 1H, arom.-H), (8.25 singlet, 1H, -CH=N). ESI-Mass calcd. (found) for $C_{19}H_{19}N_5O_4S$: 413.12 (414.15).

RESULTS AND DISCUSSION

As an aspect of continuing research to explore new biotic active compounds, a sequence of novel analogues of (*E*)-2-benzylidene-*N*-(3-(3-oxo-2,3-dihydro-4*H*-benzo[*b*][1,4]-oxazin-4-yl)propyl)hydrazine-1-carbothioamide derivatives (**9a-j**) have been synthesized and characterized. The 4*H*-benzo-[1,4]oxazine-3-one (**3**) can be synthesized in excellenet yield by the reaction of 2-aminophenol (**1**) with 2-chloroacetyl chloride (**2**) in presence of benzyltriethylammonium chloride (TEBA) and $NaHCO_3$ in $CHCl_3$ following a known procedure, which then treated with 1,3-dibromo propane (**4**) in presence of K_2CO_3 and dry DMF to obtain 4-(3-bromo-propyl)-2*H*-benzo[*b*][1,4]-oxazin-3(4*H*)-one (**5**) light coloured yellowish solid with 90% yield. In LC-MS the mass to formal charge ratio = 270 [M+H], in infrared spectrum emergence about 1,4-benzoxinone C=O band at 1762 cm^{-1}. The ^{1}H NMR revealed the multiplet at δ

2.15-2.25 corresponding to methyl protons (-CH-), one triplet at δ 3.42 ppm correspond to -CH_2-Br and another triplet at δ 4.05 ppm corresponds to -CH_2-N, -OCH_2-CO- confirmed by a sharp singlet at δ 4.55 and 6.90-7.08 ppm multiplet correlated to the aromatic four protons confirmed the bromo-alkylated derivative **5**.

Required (*Z*)-*N*′-benzylidene hydrazine carbothioamides (**8a-j**) appeared to be in very good product formation by the condensation of thiosemicarbazide (**7**) and with various benzaldehydes (**6a-j**) in presence of 2:1 ratio of ethanol:H_2O at room temperature following well-established literature procedure.

Finally, the targeted (*E*)-2-benzylidene-*N*-(3-(3-oxo-2,3-dihydro-4*H*-benzo[*b*][1,4]oxazin-4-yl)propyl)hydrazine-1-carbothioamide derivatives (**9a-j**) had been achieved in excellent yield (68-80%) when (*Z*)-*N*′-benzylidene hydrazine carbothioamides (**8a-j**) reacted with 4-(3-bromopropyl)-2*H*-benzo[*b*]-[1,4]oxazin-3(4*H*)-one (**5**) in K_2CO_3 and dry *N,N*-dimethyl formamide.

All the new synthesized derivatives **9a-j** was characterized using various spectroscopic methods. Spectrograph results about methodized designs be in sufficient harmony with the suggested frameworks. For instance, compound **9b** was characterized by its ^{1}H NMR spectrum, which showed characteristic -N-CH_2- protons of propyl hydrazine-1-carbothioamide coupled 1,4-benzoxazine seemed as a triplet at δ 4.00-4.13 ppm; -$NHCH_2$- protons emerged as a triplet at δ 3.11-3.20 ppm and -OCH_2- of 1,4-benzoxazine ring at δ 4.60 ppm, which was further confirmed by LC-MS having a mass *m/z* = 437.15 [M+H] of $C_{20}H_{19}N_4O_2SF_3$. Moreover, the IR spectra for the compounds **9a-j** indicated distinctive absorption bands at 3441, 2975, 1727, 1664, 1588 cm^{-1}, which correlated to -NH, C-H, C=O, C=N and C-N, individually.

Conclusion

A new series of (*E*)-2-benzylidene-*N*-(3-(3-oxo-2,3-dihydro-4*H*-benzo[*b*][1,4]oxazin-4-yl)propyl)hydrazine-1-carbothioamide (**9a-j**) had been accomplished in moderate to excellent yield (68-80%) when (*Z*)-*N*′-benzylidene hydrazine carbothioamides (**8a-j**) reacted with 4-(3-bromopropyl)-2*H*-benzo[*b*]-[1,4]oxazin-3(4*H*)-one. The synthesized compounds were confirmed ^{1}H NMR, IR and mass spectral analysis.

ACKNOWLEDGEMENTS

The authors are thankful to the Department of Pharmacy Chaitanya Deemed to be University, Hanamkonda, India for providing the necessary research facilities.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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