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Research article

Synthesis and antimicrobial evaluation of some new benzthiazole oxime ether derivatives

Sachin V Patil*[1], Deepak R Nagergoje[2], Arun G Dholi[1], Manjusha B Suryavanshi[1]

1. HPT Arts and RYK Science College, Nashik, Maharashtra, India
2. Arts, Science and Commerce College, Mokhada, Maharashtra, India

ABSTRACT

Some new oxime ether derivatives containing benzthiazole heterocyclic nuclei are synthesized. The reaction of 2-mercapto benzthiazole with α-halo ketones followed by reaction with hydroxylamine gave oxime derivatives which on reaction with alkyl halides viz. ethyl chloride, n-propyl chloride, and n-butyl chloride in absolute ethanol afforded the target compounds 4a-l. The structure of all the synthesized compounds was confirmed by spectroscopic methods like mass and NMR. All compounds after structural confirmation were tested for biological activities.

Keywords: Oxime ether derivatives, 2-mercapto benzthiazole, α-halo ketones, Alkyl halides, Biological activity

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Correspondence: Dr.Sachin Vishnu Patil* ✉ sachin.dhokare@yahoo.com

Research Institute, Department of Chemistry, HPT Arts and RYK Science College, Nashik-422005, Maharashtra, India

INTRODUCTION

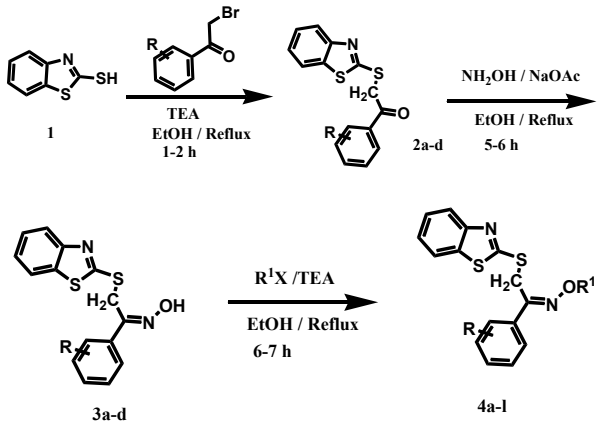
Oxime ether derivatives containing molecules have attracted significant consideration in agrochemical and medicinal research, due to excellent bioactivities such as insecticidal, [1, 2] fungicidal, [3-9] herbicidal, [10,11] another antitumor, [12-16] antiphytoviral, [17] acaricidal, [18] antiviral, [19] anticonvulsant, [20] antibacterial, and ectoparacidal activity.[21] Among the important heterocyclic nuclei, benzthiazole occupy a distinct place in drug discovery research. This is an important group of heterocycles that are substructures of many drug molecules. Derivatives of these heterocycles are an important part of therapeutic agents like anti-microbial, anti-viral, antihistaminic, anticonvulsant, antidepressant, and anti-tumor activity. Till today very little research has been done on oxime ether derivatives of benzthiazole derivatives. In view of the above considerations, the design and synthesis of newer antimicrobials remain an area of immense significance. This work deals with the synthesis of novel oxime ether molecules containing benzthiazole heterocyclic nuclei.

EXPERIMENTAL WORK

In this section, synthesis of new oxime ether derivatives containing benzthiazole heterocycle 4a-l is reported. The synthesized products were tested for biological activities against different bacteria and fungi shown in Table 1. The target molecules were synthesized as per the Scheme1. Reaction of 2-mercapto benzthiazole with α-halo ketones afforded S-substituted benzthiazole derivatives 2a-d.

Compounds 2a-d was further converted to their respective oxime derivatives 3a-d by reaction with hydroxyl amine hydrochloride and sodium acetate in ethanol. The oxime derivatives were then refluxed with different alkyl halides viz. ethyl chloride, n-propyl chloride and n-butyl chloride in absolute ethanol to obtain final products 4a-l.

Scheme I: Synthetic route for 4a-l



RESULT AND DISCUSSION

The structure of the intermediates, 2a-d was confirmed by Spectral analysis. Signal in IR at 1690-1700 cm^{-1} is because of presence of C=O group. The formation of the oxime was also

confirmed by disappearance of band at 1690-1700 cm^{-1} and appearance of new band at 3200-3300 cm^{-1}, 1580-1590 cm^{-1}, owing to C=N and OH groups correspondingly. The oxime derivatives 3a-d was also confirmed by ^1H NMR. All the spectra showed broad singlet at 8.3-8.4 ppm for OH proton, a singlet at 4.1-4.6 ppm for CH_2 protons while the multiplet at 7.3-8.00 ppm was assigned to aromatic protons. The disappearance of broad singlet at 8.3-8.4 ppm for OH proton in ^1H NMR confirmed the conversion of oxime to its oxime ether derivative 4a-l. The o-alkylation reaction confirmation was identified from IR which showed absence of signal 3200-3300 cm^{-1}. The IR bands at 1580-90 cm^{-1} and 1000-1050 cm^{-1} are due to CN and CO groups correspondingly. The formation of compounds 4a-l was also identified by other spectroscopic techniques like elemental analysis, NMR and Mass. The isotopic peaks in mass are helpful to detect the presence of Chlorine and Bromine in the molecules.

BIOLOGICAL RESULTS AND DISCUSSION

All the final products were identified for biological activities. The results are shown in Table 1. It was found that all compounds showed good activities against fungi species, whereas less active against bacteria. Compounds with bromine substituent showed good antifungal activities (4d, 4e and 4f) whereas in case of compounds which contain NO_2 group either at meta or para position 4g-l showed decreased antifungal activity.

Table 1: Biological activities of final products 4a-l

Compd.	S. aureus	E. coli	B. subtilis	P. aeruginosa	A. niger	C. albicans
4a	5.81	-	6.65	-	13.22	14.5
4b	-	5.10	-	5.76	-	13.55
4c	4.23	-	6.11	5.65	10.52	11.00
4d	7.52	6.75	-	9.5	15.22	16.50
4e	-	6.00	-	-	-	14.35
4f	6.32	-	7.12	-	14.42	-
4g	4.89	4.00	-	7.30	-	9.75
4h	2.55	1.66	-	4.56	10.15	-
4i	1.65	-	2.11	3.38	10.00	9.84
4j	5.20	3.11	-	-	-	-
4k	4.50	-	-	4.20	11.00	10.72
4l	4.11	2.25	3.55	-	10.51	9.05
Nystatin	NA	NA	NA	NA	21.12	21.96
Chloram - phenicol	32.8	29.14	30.11	24.68	NA	NA

Chloramphenicol (100 µg/disc), Nystatin (100 µg/disc) reference; conc. of compound 4a-l (100 µg/disc)

EXPERIMENTAL

General method for preparation of 2-(benzo[d]thiazol-2-ylthio)-1-aryl-ethanone (2a-d)

2-mercaptobenzthiazole (10 mmol), α-haloketone (10 mmol) and triethyl amine (0.01 mmol) in round bottom was refluxed in ethanol for one hour. TLC technique used to detect the reaction progress using proper solvent. After completion of reaction, solvent was removed and the residual product was purified from ethanol solvent to get target compound 2a-d.

General method for preparation of 2-(benzo[d]thiazol-2-ylthio)-1-aryl-ethanone oxime derivatives (3a-d)

Above synthesized compounds 2a-d (10 mmol), hydroxylamine hydrochloride (10 mmol) and sodium acetate (20 mmol) in ethanol was refluxed for 3-4 hours. The reaction mixture was then poured in cold water to obtain the precipitate of the products 3a-d, which was purified by crystallization from ethanol with good yields.

General method for preparation of o-alkyl oxime derivatives (4a-l)

Above synthesized products 3a-d(0.015 mol), alkyl halide (0.015 mol) and triethyl amine (0.001 mol) wasrefluxed for 6-7 hours in ethanol. The reaction mixture was then poured in water and extracted in ethyl acetate. The solvent was then removed to get products 4a-l which were purified by column chromatography.

SPECTRAL DATA

4a)2-(benzo[d]thiazol-2-ylthio)-1-(4chlorophenyl)ethanone-o-ethyl-oxime: Yield: (59 %); mp: 52-55°C; IR (KBr, cm^{-1}): 2940, 1110, 1585 (C=N), 980 (N-O); ^1H NMR (300 MHz, $CDCl_3$): δ 7.3-7.8(m, 4H, Ar-H), 4.8(s, 2H, S-CH_2), 7.6(d, J = 8.3 Hz, 2H, Ar-H), 7.4(d, J = 8.3 Hz, 2H, Ar-H), 4.3(q, J = 6.6 Hz, 2H, O-CH_2,) 1.3(t, J = 6.6 Hz, 3H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$): δ 135.2, 122.1, 126.5, 125.3, 122.3, 151.0, 152.2, 31.0(S-CH_2), 164.3, 72.2(O-CH_2), 24.2(CH_3), 133.4, 130.3(2C), 129.2(2C), 131.3; Anal. Calcd for $C_{17}H_{15}ClN_2OS_2$: C, 56.26; H, 4.17; N, 7.72; Found: C, 56.19; H, 4.09; N, 7.64; ms: m/z 362.01(M+), 364.02(M+2).

4b) 2-(benzo[d]thiazol-2-ylthio)-1-(4-chlorophenyl)ethanone-o-proyl-oxime: Yield: (55 %); mp: 48-54°C; IR (KBr, cm^{-1}): 3010, 1120, 1600 (C=N), 990 (N-O) cm^{-1}, ^1H NMR (300 MHz, $CDCl_3$): δ 7.3-7.8(m, 4H, Ar-H), 4.8(s, 2H, S-CH_2), 7.6(d, J = 8.3 Hz, 2H, Ar-H), 7.4(d, J = 8.3 Hz, 2H, Ar-H), 4.3(t, J = 6.7 Hz, 2H, O-CH_2,), 1.6(m, 2H, CH_2), 1.0(t, J = 6.8 Hz, 3H, CH_3); Anal. Calcd. for $C_{18}H_{17}ClN_2OS_2$: C, 57.36; H, 4.55; N, 7.43; Found: C, 57.28; H, 4.49; N, 7.37; ms: m/z 376.07(M+), 378.03(M+2).

4c)2-(benzo[d]thiazol-2-ylthio)-1-(4-chlorophenyl)ethanone-o-nbutyl-oxim:Yield: (58 %); mp: 62-65°C; ^1H NMR (300 MHz, $CDCl_3$): δ 7.5-7.8(m, 4H, Ar-H), 4.8(s, 2H, S-CH_2), 7.6(d, J = 8.4 Hz, 2H, Ar-H), 7.4(d, J = 8.4 Hz, 2H, Ar-H), 4.3(t, J = 6.6 Hz, 2H, O-CH_2), 1.5-1.6(m, 4H, $2CH_2$), 0.9(t, J = 6.7 Hz, 3H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$): δ 135.1, 122.1, 126.2, 125.3, 122.5, 151.0, 151.7, 30.8(S-CH_2), 163.8, 72.2(O-CH_2), 20.4(CH_2), 18.5(CH_2), 14.2(CH_3), 133.4, 130.5(2C), 129.1(2C), 131.3; Anal.Calcd. for $C_{19}H_{19}ClN_2OS_2$: C, 58.37; H, 4.90; N, 7.17; Found: C, 58.29; H, 4.80; N, 7.24; ms: m/z 390.03(M+), 392.07(M+2).

4d) 2-(benzo[d]thiazol-2-ylthio)-1-(4-bromophenyl)ethanone-o-ethyl-oxime:Yield: (61%); mp: 82-85°C; IR (KBr, cm^{-1}): 3000, 1130, 1550 (C=N), 980 (N-O); ^1H NMR (300 MHz, $CDCl_3$): δ 7.3-7.8(m, 4H, Ar-H), 4.8(s, 2H, S-CH_2), 7.6(d, J = 8.4 Hz, 2H, Ar-H), 7.4(d, J = 8.4

Hz, 2H, Ar-H), 4.2(q, J = 6.5 Hz, 2H, O-CH_2) 1.2(t, J = 6.5 Hz, 3H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$): δ 135.5, 121.7, 126.4, 125.5, 122.3, 152.1, 153.3, 31.1(S-CH_2), 165.0, 70.0(O-CH_2), 24.0(CH_3), 134.4, 131.3(2C), 132.0(2C), 124.4; Anal. Calcd for $C_{17}H_{15}BrN_2OS_2$: C, 50.12; H, 3.71; N, 6.88; Found: C, 50.04; H, 3.80; N, 6.71; ms: m/z 406.03(M+), 408.03(M+2).

4e) 2-(benzo[d]thiazol-2-ylthio)-1-(4-bromophenyl)ethanone-o-proyl-oxime: Yield: (66 %); mp: 102-105°C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.3-7.8(m, 4H, Ar-H), 4.8(s, 2H, S-CH_2), 7.6(d, J=8.4 Hz, 2H, Ar-H), 7.4(d, J= 8.4 Hz, 2H, Ar-H), 4.3(t, J = 6.6 Hz, 2H, O-CH_2), 1.5(m, 2H, CH_2), 1.0(t, J = 6.7 Hz, 3H, CH_3); Anal. Calcd for $C_{18}H_{17}BrN_2OS_2$: C, 51.31; H, 4.07; N, 6.65; Found: C, 51.26; H, 4.10; N, 6.54; ms: m/z 420.02(M+), 422.06(M+2).

4f)2-(benzo[d]thiazol-2-ylthio)-1-(4-bromophenyl)ethanone-o-n-butyl-oxime: Yield: (61 %); mp: 95-101°C; IR (KBr, cm^{-1}): 3020, 1110-1130, 1560 (C═N), 980 (N-O); ^{1}H NMR (300 MHz, CDCl3): δ 7.3-7.8(m, 4H, Ar-H), 4.8 (s, 2H, S-CH_2), 7.6(d, J = 8.4 Hz, 2H, Ar-H), 7.4(d, J = 8.4 Hz, 2H, Ar-H), 4.3(t, J = 6.6 Hz, 2H, O-CH_2), 1.3-1.5(m, 4H, 2CH_2), 0.8(t, J = 6.8 Hz, 3H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$): δ135.0, 121.5, 126.2, 125.3, 122.1, 151.0, 152.1, 30.3(S-CH_2), 164.6, 71.0(O-CH_2), 20.2(CH_2), 17.5(CH_2), 14.3(CH_3), 134.1, 131.5(2C), 132.0(2C), 124.0; Anal. Calcd. for $C_{19}H_{19}BrN_2OS_2$: C, 52.41; H, 4.40; N, 6.43; Found: C, 52.32; H, 4.33; N, 6.35; ms: m/z 434(M+), 436.04(M+2).

4g) 2-(benzo[d]thiazol-2-ylthio)-1-(4-nitrophenyl)ethanone-o-ethyl-oxime:Yield: (57 %); mp: 70-73°C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.3-7.8(m, 4H, Ar-H), 4.8(s, 2H, S-CH_2), 8.2(d, J = 8.2 Hz, 2H, Ar-H), 8.1(d, J = 8.2 Hz, 2H, Ar-H), 4.3(q, J = 6.7Hz, 2H, O-CH_2), 1.1(t, J = 6.7Hz, 3H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$): δ 135.8, 121.5, 126.0, 125.0, 121.8, 151.8, 153.3, 30.0(S-CH_2), 165.5, 71.5(O-CH_2), 25.0(CH_3), 140.2, 127.8(2C), 123.7(2C), 148.4;Anal. Calcd for $C_{17}H_{15}N_3O_3S_2$: C, 54.67; H, 4.05; N, 11.25; Found: C, 54.59; H, 4.11; N, 11.19; ms: m/z 373(M+), 375(M+2).

4h) 2-(benzo[d]thiazol-2-ylthio)-1-(4-nitrophenyl)ethanone-o-proyl-oxime: Yield: (61 %); oil: IR (KBr, cm^{-1}): 3010, 1100, 1550 (C═N), 985 (N-O) cm^{-1}, 1345. 1335; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.3-7.8(m, 4H, Ar-H), 4.8(s, 2H, S-CH_2), 8.2(d, J = 8.2 Hz, 2H, Ar-H), 8.1(d, J = 8.2 Hz, 2H, Ar-H), 4.3(t, J = 6.65 Hz, 2H, O-CH_2), 1.4(m, 2H, CH_2), 0.9(t, J = 6.7 Hz, 3H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$): δ 135.5, 121.2, 126.0, 125.2, 122.3, 151.8, 153.0, 30.0(S-CH_2), 165.6, 71.0(O-CH_2), 20(CH_2), 15.6(CH_3), 140.4, 127.8(2C), 123.7(2C), 148.0; Anal. Calcd. for $C_{18}H_{17}N_3O_3S_2$: C, 55.80; H, 4.42; N, 10.84; Found: C, 55.71; H, 4.34; N, 10.76; ms: m/z 387.04(M+), 389.05(M+2).

4i)2-(benzo[d]thiazol-2-ylthio)-1-(4-nitrophenyl)ethanone-o-n-butyl-oxime: Yield: (59 %); mp: 52-56°C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.3-7.7(m, 4H, Ar-H), 4.7(s, 2H, S-CH_2), 8.2(d, J = 8.3 Hz, 2H, Ar-H), 8.1(d, J = 8.3 Hz, 2H, Ar-H), 4.3(t, J = 6.6 Hz, 2H, O-CH_2), 1.3-1.5(m, 4H, 2CH_2), 0.89(t, J = 6.7 Hz, 3H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$): δ 135.0, 121.4, 126.4, 125.1, 122.2, 151.8, 153.0, 30.0(S-CH_2), 165.5, 70.0(O-CH_2), 20.0(CH_2), 17.5(CH_2), 13.5(CH_3), 140.3, 127.5(2C), 123.4(2C), 147.5; Anal. Calcd. for $C_{19}H_{19}N_3O_3S_2$: C, 56.84; H, 4.77; N, 10.47; Found: C, 56.77; H, 4.62; N, 10.35; ms: m/z 401.1(M+), 403.07(M+2).

4j)2-(benzo[d]thiazol-2-ylthio)-1-(3-nitrophenyl)ethanone-o-ethyl-oxime: Yield: (55 %); mp: 115-120°C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.3-7.8(m, 4H, Ar-H), 4.8(s, 2H, S-CH_2), 8.1(d, J = 8.4 Hz, 1H, Ar-H), 7.5(m, 1H, Ar-H), 8.2(d, J = 8.5 Hz, 1H, Ar-H), 8.5(s, 1H, Ar-H), 4.3(q, J = 6.68 Hz, 2H, O-CH_2),1.35(t, J = 6.68 Hz, 3H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$): δ 135.0, 122.0, 125.2, 124.5, 122.3, 151.0, 152.3, 30.4(S-CH_2), 165.0, 71.2(O-CH_2), 25.8(CH_3), 134.7, 131.4, 127.4, 124.3, 144.2, 123.3; Anal. Calcd. for $C_{17}H_{15}N_3O_3S_2$: C, 54.67; H, 4.05; N, 11.25; Found: C, 54.59; H, 4.11; N, 11.18; ms: m/z 373.08(M+), 375.05(M+2).

4k) 2-(benzo[d]thiazol-2-ylthio)-1-(3-nitrophenyl)ethanone-o-proyl-oxime: Yield: (65 %); mp: 108-113°C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.3-7.8(m, 4H, Ar-H), 4.8(s, 2H, S-CH_2), 8.1(d, J = 8.4 Hz, 1H, Ar-H), 7.5(m, 1H, Ar-H), 8.2(d, J = 8.4 Hz, 1H, Ar-H), 8.5(s, 1H, Ar-H), 4.3(t, J = 6.6 Hz, 2H, O-CH_2), 1.5(m, 2H, CH_2), 0.9(t, J = 6.9 Hz, 3H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$): δ 135.3, 122.2, 125.1, 124.3, 122.6, 151.0, 152.3, 30.4(S-CH_2), 164.7, 71.0(O-CH_2), 22.2(CH_2), 14.2(CH_3), 134.7, 131.3, 127.4, 123.6, 144.0, 123.5; Anal. Calcd. for $C_{18}H_{17}N_3O_3S_2$: C, 55.80; H, 4.42; N, 10.84; Found: C, 55.71; H, 4.30; N, 10.73; ms: m/z 387.07(M+), 389.01(M+2).

4l)2-(benzo[d]thiazol-2-ylthio)-1-(3-nitrophenyl)ethanone-o-n-butyl-oxime: Yield: (63 %); mp: 125-130°C; IR (KBr, cm^{-1}): 3015, 1125, 1580 (C═N), 1350, 1545, 980 (N-O); ^{1}HNMR (300 MHz, $CDCl_3$): δ 7.3-7.8(m, 4H, Ar-H), 4.8(s, 2H, S-CH_2) ,8.2(d, J=8.3 Hz, 1H, Ar-H), 7.6(m, 1H, Ar-H), 8.2(d, J = 8.3 Hz, 1H, Ar-H), 8.4(s, 1H, Ar-H), 4.3(t, J = 6.5 Hz, 2H, O-CH_2), 1.3-1.4(m, 4H, 2CH_2), 0.8(t, J = 6.8 Hz, 3H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$): δ 135.0, 122.3, 125.3, 124.2, 122.1, 151.5, 152.0, 30.0(S-CH_2), 165.0, 71.0(O-CH_2), 20.2(CH_2), 17.5(CH_2), 13.3(CH_3), 134.5, 131.4, 127.4, 124.1, 144.0, 123.2; Anal. Calcd. for $C_{19}H_{19}N_3O_3S_2$: C, 56.84; H, 4.77; N, 10.47; Found: C, 56.78; H, 4.68; N, 10.39; ms: m/z 401.05(M+), 403.07 (M+2).

CONCLUSION

In conclusion, O-alkyl derivatives of benzthiazole nuclei were planned and all were prepared successfully. It was detected that all the final compounds 4a-l revealed decent to moderate biological activities against different bacteria and fungi. From this study, we can conclude that oxime ether derivatives containing benzthiazole nuclei could be a good model to synthesize new antifungal drugs.

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