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Synthesis and Antimicrobial Activity of Cyclic Dithiocarbamates Employing Triton-B/CS_2 System

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An efficient and green methodology to synthesize cyclic dithiocarbamates (compounds **1-11**) was developed by reaction of primary amines, CS_2 and 1,2-dibromoethane or 1,4-dibromobutane, catalyzed by Triton-B (as PTC)/CS_2 system. Mass spectroscopy, elemental analysis and ^{1}H NMR are used for characterization of the synthesized compounds (**1-11**). This effectual green tactics give good yield of product which entails mild conditions. Compounds **(1-11)** were found to possess *in vitro* antimicrobial activities against the pathogenic bacterial and fungal strain. The microbial strains used to screen activities are *Escherichia coli*, *Staphylococcus aureus* and *Candida albicans.*

Keywords: Primary amine, Dithiocarbamates, Dihaloalkane, Triton-B, Biological activities.

INTRODUCTION

Cyclic dithiocarbamates are important class of organo-sulfur compounds that display plethora of important applications such as pharmaceuticals and agrochemicals [1-4], insecticide and worldwide as crop protection pest controls (thiacloprid) [5], numerous analogous [6], antiproliferative [7], hypertension and hyperlipidemia [8], analgesic and antiparkinsonian activity [9], antioxidants [10], transient receptor potential vanilloid 1 (TRPV-1) [11], pesticides [12], central nervous systems [13], anti-inflammatory (COX-2 inhibitor) and ulcerogenic activity [14,15], antibacterial [16], antidiabetic [17], antifungal [18], anti-HIV [19], oncological activity [20], antitumor [21], tyrosine inhibitor [22], antibiofilm [23], anticonvulsant [24], wound healing [25], phyto-growth inhibitory [26], antihyperglycemic [27], fungicidal activity [28] and group transfer radical cyclization reactions [29], *etc.* Furthermore, transition metals complexes of dithiocarbamates have been stated for many reviews since they are used as organic superconductors [30]. Organic cyclic dithiocarbamates had been used for facile and versatile synthesis [31-41] and well-thought-out as significant biological moiety. Cyclic dithiocarbamates are evaluated for their toxicological activity [42]. Traditionally, toxic chemicals such as phosgene, thiophosgene and its byproducts are used to prepare organic cyclic dithiocarbamates but this is time taking method and require high temperature. Different effectual green methods are designated in literature to produce cyclic dithiocarbamates. In recent years, our research group has been realized that benzyl trimethylammonium hydroxide (Triton-B) has been used even as emerging catalyst for facile synthesis of numerous organic compounds [43-47]. We have also synthesized various kinds of dithiocarbamate employing CS_2/Triton-B system from variety of starting materials [48]. In this article, thiazolidine-2-thiones were synthesized starting with the corresponding dihaloalkane, 1º amines employing Triton-B/CS_2 system and the synthesized compounds have been assessed by antimicrobial activity.

EXPERIMENTAL

Chemicals bought from Alfa-Aesar, GLR, Lobachem, AVRA and Finar, Labchem. MB-104-FTIR spectrophotometer of ABB Bomem, was used for recording infrared spectra (4000-200cm^{-1}). The AC-400F-nuclear magnetic resonance spectrometer with tetramethyl silane as internal standard was used for recording ^{1}H NMR spectra at 400 MHz. The examinations of elements were conveyed with the help of a 1110-CNNO-S (Carlo-ErbaEA) analyzer.

General protocol: Primary amine (1 mmol) in DMSO and 10 mmol of CS_2 was stirred for 15 min after adding 3 mmol of Triton-B as phase transfer catalyst.The reaction mass was again stirred for 10 min after adding (1,2-dibromoethane or 1,4-dibromobutane) dropwise under argon atmosphere. The reacting mixture was stirred at room temperature for 1-2 h. The reaction mixture was monitored by TLC. After completion the reaction mass was quenched with 50 mL distilled H_2O and pull out with ethyl acetate thrice. The layer of organic compound was separated and dried by anhydrous Na_2SO_4 and concentrated to get the crude compound. Column chromatography was used to purify crude compound, using silica-gel (100-200 mesh) and eluent 20% (ethylacetate: hexane) to give the desired product.

3-Phenyl-1,3-thiazepane-2-thione (1): Yellow solid, m.p.: 62 ºC. Elemental analysis of $C_{11}H_{13}NS_2$ calcd. (found) (%): C, 59.15 (59.35); H, 5.87 (5.95); N, 6.27 (6.32); S, 28.71 (28.95). ^{1}H NMR (400 MHz, $CDCl_3$, δ ppm): 7.429-7.317 (m, 5H), 3.328 (m, 4H), 1.824 (m, 4H). MS (ESI): *m/z* $(M)^+$ calculated = 223.05, Found $(M+1)^+$ = 223.36.

3-Heptyl-1,3-thiazepane-2-thione (2): Yellow solid, m.p.: 67 ºC. Elemental analysis of $C_{12}H_{23}NS_2$ calcd. (found) (%): C, 58.72 (58.95); H, 9.45 (9.65); N, 5.71 (5.85); S, 26.13 (26.45). ^{1}H NMR (400 MHz, $CDCl_3$, δ ppm): 3.348 (t, *J* = 7.2 Hz, 2H), 3.137 (d, *J* = 5.6 Hz, 2H), 3.017 (d, *J* = 5.6 Hz, 2H), 2.256-2.210 (m, 4H), 1.654 (m, 2H), 1.425-1.213 (m, 8H), 0.875 (t, *J* = 6.4 Hz, 3H). MS (ESI): *m/z* $(M)^+$ calculated = 245.15, Found $(M+1)^+$= 245.45.

3-(2,4-Dichlorobenzyl)-1,3-thiazepane-2-thione (3): Yellow solid, m.p.: 121 ºC. Elemental analysis of $C_{12}H_{13}NS_2Cl_2$ calcd. (found) (%): C, 47.06 (47.25); H, 4.28 (4.32); Cl, 23.15 (23.35); N, 4.57 (4.87); S, 20.94 (21.14). ^{1}H NMR (400 MHz, $CDCl_3$, δ ppm): 7.462-7.385 (m, 2H), 7.260 (s, 1H), 4.984 (t, *J* = 5.6 Hz, 2H), 4.173 (s, 2H), 3.429 (q, *J* = 6.8 Hz, 2H), 3.305 (q, *J* = 6.8 Hz, 2H), 3.159 (t, *J* = 5.6 Hz, 2H). MS (ESI): *m/z* $(M)^+$ calculated = 304.99, Found $(M+1)^+$= 306.27.

3-(2,4-Dichlorobenzyl)thiazolidine-2-thione (4): Yellow solid, m.p.: 93 ºC. Elemental analysis of $C_{10}H_9NS_2Cl_2$ calcd. (found) (%): C, 43.17 (43.43); H, 3.26 (3.45); Cl, 25.49 (25.58); N, 5.03 (5.13); S, 23.05 (23.45). ^{1}H NMR (400 MHz, $CDCl_3$, δ ppm): 7.453 (d, *J* = 8.0 Hz, 1H), 7.368 (s, 1H), 7.230 (t, *J* = 5.6 Hz, 2H), 3.466 (t, *J* = 5.6 Hz, 2H). MS (ESI): *m/z* $(M)^+$ calculated = 276.96, Found $(M+1)^+$ = 278.22.

3-(3-Fluorobenzyl)thiazolidine-2-thione (5): Yellow solid, m.p.: 98 ºC. Elemental analysis of $C_{10}H_{10}NS_2F$ calcd. (found) (%): C, 52.84 (52.94); H, 4.43 (4.53); F, 8.36 (8.52); N, 6.16 (6.35); S, 28.21 (28.43). ^{1}H NMR (400 MHz, $CDCl_3$, δ ppm): 7.626-7.308 (m, 2H), 7.129-7.026 (m, 3H), 4.926 (s, 2H), 3.957 (t, *J* = 8.4 Hz, 2H), 3.263 (t, *J* = 8.0 Hz, 2H). MS (ESI): *m/z* $(M)^+$ calculated = 227.02, Found $(M+1)^+$ = 227.32.

3-(3-Fluorobenzyl)-1,3-thiazepane-2-thione (6): Yellow solid, m.p.: 98 ºC. Elemental analysis of $C_{12}H_{14}NS_2F$ calcd. (found) (%): C, 56.44 (56.62); H, 5.53 (5.68); F, 7.44 (7.72); N, 5.48 (5.92); S, 25.11 (25.98). ^{1}H NMR 400 MHz, $CDCl_3$, δ ppm: 7.337-7.280 (m, 2H), 7.207-6.975 (m, 3H), 4.929 (s, 2H), 3.440 (t, *J* = 6.4 Hz, 2H), 3.389-3.268 (m, 4H), 3.150 (t, *J* = 5.2 Hz, 2H). MS (ESI): *m/z* $(M)^+$ calculated = 255.06, Found $(M+1)^+$ = 255.36.

3-(3-Fluorophenylethyl)-1,3-thiazepane-2-thione (7): Yellow solid, m.p.: 82 ºC. Elemental analysis of $C_{13}H_{16}NS_2F$ calcd. (found) %: C, 57.96 (58.26); H, 5.99 (6.56); F, 7.05 (7.35); N, 5.20 (5.43); S, 23.80 (23.98).^{1}H NMR (400 MHz, $CDCl_3$, δ ppm): 7.519 (s, 1H), 7.038-6.914 (m, 3H), 3.966 (q, *J* = 6.8 Hz, 2H), 3.392-3.283 (m, 4H), 2.978 (t, *J* = 6.4 Hz, 2H), 1.834-1.784 (m, 4H). MS (ESI): *m/z* $(M)^+$ calculated = 269.06, Found $(M+1)^+$ = 269.40.

3-(4-Methoxy-phenylethyl)-1,3-thiazepane-2-thione (8): Yellow solid, m.p.: 82 ºC. Elemental analysis of $C_{13}H_{16}NS_2F$ calcd. (found) %: C, 59.75 (59.86); H, 6.80 (6.96); N, 4.98 (5.45); O, 5.68 (5.79), S, 22.79 (22.98). ^{1}H NMR (400 MHz, $CDCl_3$, δ ppm): 7.125 (d, *J* = 8.0 Hz, 2H), 6.876 (d, *J* = 8.4 Hz, 2H), 3.955 (q, *J* = 6.4 Hz, 2H), 3.798 (s, 3H), 3.390-3.267 (m, 4H), 2.909 (t, *J* = 6.8 Hz, 2H), 1.820-1.770 (m, 4H). MS (ESI): *m/z* $(M)^+$ calculated = 281.09, Found $(M+1)^+$= 281.44.

3-(2,4-Difluorobenzyl)thiazolidine-2-thione (9): Yellow solid, m.p.: 87 ºC. Elemental analysis of $C_{10}H_9NS_2F_2$ calcd. (found) %: C, 48.96 49.13); H, 3.70 (3.89); F, 15.49 (15.75); N, 5.71 (5.91); S, 26.14 (26.64). ^{1}H NMR (400 MHz, $CDCl_3$, δ ppm): 7.410 (t, *J* = 7.2 Hz, 1H), 7.128 (s, 1H), 6.883-6.814 (m, 2H), 4.935 (s, 2H), 3.361 (t, *J* = 8.0 Hz, 2H), 1.799 (t, *J* = 8.0 Hz, 2H). MS (ESI): *m/z* $(M)^+$ calculated = 245.01, Found $(M+1)^+$ = 245.31.

3-(3-Bromo-phenylethyl)-1,3-thiazepane-2-thione (10): Yellow solid, m.p.: 64 ºC. Elemental analysis of $C_{13}H_{16}NS_2Br$, calcd. (found) %: C, 47.27 (47.52); H, 4.88 (4.98); Br, 24.19 (24.59); N, 4.24 (4.98); S, 19.42 (19.73). ^{1}H NMR (400 MHz, $CDCl_3$, δ ppm): 7.632-7.369 (m, 2H), 7.198 (t, *J* = 7.6 Hz, 1H), 7.151 (d, *J* = 7.6 Hz, 1H), 3.968 (q, *J* = 6.8 Hz, 2H), 3.393 (m, 4H), 2.974 (t, *J* = 6.8 Hz, 2H), 1.821-1.788 (m, 4H). MS (ESI): *m/z* $(M)^+$ calculated = 328.99, Found $(M+1)^+$= 330.31.

3-(2-Methylbenzyl)thiazolidine-2-thione (11): Yellow solid, m.p.: 67 ºC. Elemental analysis of $C_{11}H_{13}NS_2$ calcd. (found) (%): C, 59.15 (59.43); H, 5.87 (5.95); N, 6.27 (6.87); S, 28.71 (28.88). ^{1}H NMR (400 MHz) ($CDCl_3$): δ 7.442-7.310 (m, 1H), 7.223-6.392 (m, 3H), 4.464 (s, 2H), 3.620 (t, *J* = 6.4 Hz, 2H), 3.436 (t, *J* = 5.6 Hz, 2H), 2.331 (s, 3H).MS (ESI): *m/z* $(M)^+$ calculated = 223.06, Found $(M+1)^+$= 223.36.

RESULTS AND DISCUSSION

Three components primary amines (**1**), CS_2 (**2**) with 1, 2-dibromoethane or 1,4-dibromobutane (**3**) and Triton-B catalyst were reacted in DMSO at room temperature to produce thiazolidine-2-thiones or thiazepane-2-thione (**4**) to get good yields (Table-1). The CS_2 mediated synthesis is described for the synthesis of a large number of cyclic dithiocarbamates derivatives and offers some notable and distinct advantages over usually employed procedure such as simplicity, room temperature conditions, simple work-up, high yield and cheap reagent.

The synthesized compounds were found to be soluble in methanol, acetone, ethanol, ethyl acetate, dichloromethane, diethylether, DMF, $CDCl_3$, *etc*. The confirmation of product formation was done with the mass spectroscopy and^{1}H NMR. Different phase transfer catalysts (PTC) are commonly used for the synthesis but Triton-B is preferable than other PTC due to high yields of the product [49].

TABLE-1
SYNTHESIS OF THIAZOLIDINE-2-THIONE OR 1,3-THIAZEPANE-2-THIONE CATALYZED BY TRITON-B

Compd. No.	Substituted R_1	m.f.	m.w.	Yield (%)
1	Phenyl	$C_{11}H_{13}NS_2$	223.36	94.23
2	Heptyl	$C_{12}H_{23}NS_2$	245.45	95.45
3	2,4-Cl_2-benzyl	$C_{12}H_{13}NS_2Cl_2$	306.27	93.26
4	2,4-Cl_2-benzyl	$C_{10}H_9NS_2Cl_2$	278.22	93.56
5	3-F-benzyl	$C_{10}H_{10}NS_2F$	227.32	94.45
6	3-F-benzyl	$C_{12}H_{14}NS_2F$	255.37	94.58
7	3-F-phenyethyl	$C_{13}H_{16}NS_2F$	269.40	93.98
8	4-CH_3O-phenylethyl	$C_{13}H_{16}NS_2F$	281.44	94.98
9	2,4-F_2benzyl	$C_{10}H_9NS_2F_2$	245.31	93.95
10	3-Br-phenylethyl	$C_{13}H_{16}NS_2Br$	330.31	94.36
11	2-CH_3-benzyl	$C_{11}H_{13}NS_2$	223.36	90.52

The method is easier and requires simpler work-up using low cost less toxic reagents. Out-put of the product is improved and synthesized in lesser time. The compounds synthesized in this series were identified by ^{1}H NMR and mass spectra. The intermediate most probably results from addition of dithiocarbamate salt to dihaloalkane and subsequently intramolecular cyclization of the product (**Scheme-I**).

Antimicrobial activity: The derivatives were assessed for their antimicrobial activity against *Staphylococcus aureus*, *Escherichia coli* and *Candida albicans.* The antimicrobial assay was done by Agar-well diffusion method using Muller Hinton agar media for bacteria and Sabouraud Dextrose agar media for fungus. The examination was done using 1 mg/mL concentration of the synthesized derivatives in ethyl acetate as solvent. The antibiotic oxytetracycline and fluconazole were used as positive control for bacteria and fungus respectively. Some compounds showed the antimicrobial activity against the selected strain by the showing inhibition on the media. The zone of inhibition obtained is listed in Table-2.

TABLE-2
ZONE OF INHIBITION OF THE SYNTHESIZED COMPOUNDS FOR ANTIMICROBIAL ACTIVITY

Compd. No.	Substituted R_1	Zone of inhibition (mm)		
		S. aureus	*E. coli*	*C. albicans*
1	Phenyl	20	17	14
2	Heptyl	15	16	17
3	2,4-Cl_2-benzyl	20	19	14
4	2,4-Cl_2-benzyl	15	19	15
5	3-F-benzyl	18	17	14
6	3-F-benzyl	15	14	16
7	3-F-phenyethyl	12	17	14
8	4-CH_3O-phenylethyl	18	18	17
9	2,4-F_2-benzyl	12	17	15
10	3-Br-phenylethyl	18	16	17
11	2-CH_3-benzyl	20	18	15
12	(+) Control	25	22	25
13	(–) Control	Nil	Nil	Nil

Conclusion

In conclusion, a highly dexterous procedure was developed for the synthesis of cyclic dithiocabamates derivatives under the mild conditions using different primary amines with dihaloalkane *via* CS_2/Triton-B system. The derivatives formed also showed the important biological activity as some of them were found to possess the antimicrobial activity against the bacterial and fungal strain. So, these compounds may be further sight seen for their *in vivo* antimicrobial activity.

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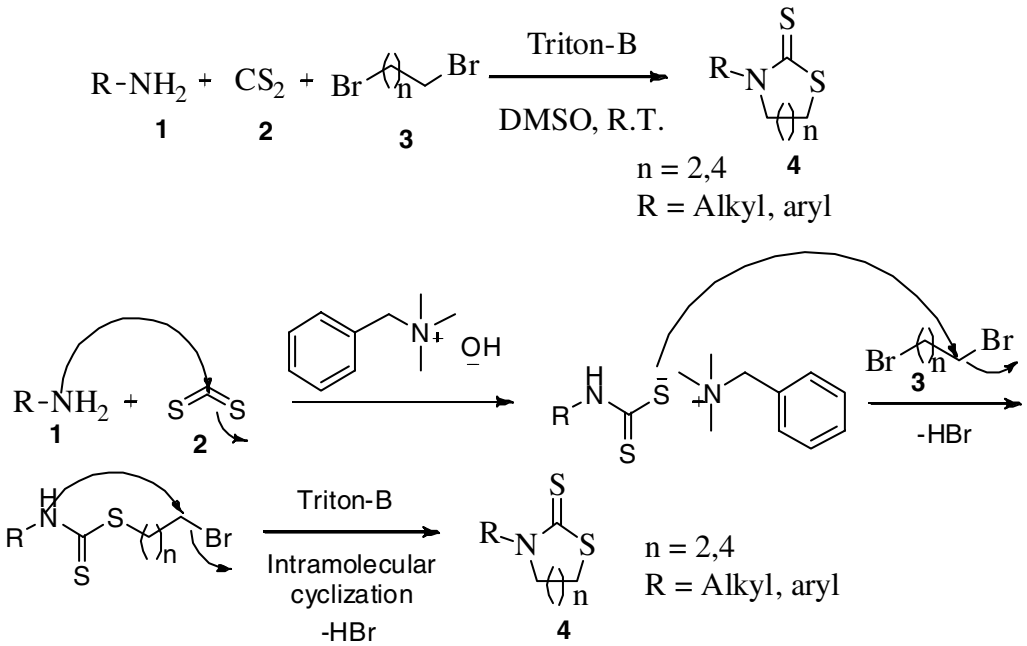

Scheme-I: Synthesis of thiazolidine-2-thione or 1,3-thiazepane-2-thione catalyzed by Triton-B

CONFLICT OF INTEREST

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

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