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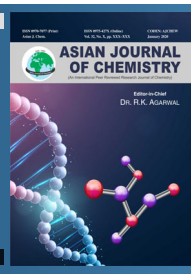

Triton-B Mediated Efficient Synthesis of Thioxoimidazolidine-4,5-diones

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A simple, mild and efficient protocol for the synthesis of thioxoimidazolidine-4,5-diones (**1-15**) from substituted thiourea employing Triton-B/CS_2 system has been discussed. The protocol described is an easy, efficient, nature-friendly method using cheap, easily available, less toxic reagents at room temperature. The synthesis involves the reaction between substituted thiourea in toluene and oxalyl chloride at room temperature using phase transfer catalyst Triton-B/CS_2 system.

Keywords: Thioxoimidazolidine-4,5-diones, Substituted thiourea, Oxalyl chloride, Toluene, Triton-B, Carbon disulfide.

INTRODUCTION

In recent years, there is a ceaseless quest for biological moiety for treating various infections [1]. The diversified illness occurring to the living being drives the demand for biologically potent scaffolds to treat such variety of illness due to increasing resistance of pathogenic bacteria towards antibiotics. So there is great demand of new antibacterial drugs [2]. The discovery of effective and least toxic products needs new synthetic routes and modification of structure of prevailing molecules using the phenomena of bioisosterism [3]. The significance of an atom or the group is analyzed on the basis of the bioactivity of the drug having similar antagonistic effect.

The 1*H*-imidazole is a five membered ring with two heteroatom, is an important scaffold found in many drugs used for microbial infection [4-7], hyperthyroidism [8] and insomnia [9]. Thioxoimidazolidine-4,5-dione are the group of heterocyclic compound with 1*H*-imidazole moiety has a wide range of biological and pharmacological properties [10,11]. 2,4-Imidazolidinones or hydantoins [3] are interesting compounds owing to their important pharmacological properties [12]. These heterocyclic moieties present in the substances make them notable bioactive substances with broad spectrum biological activities like anti-inflammatory [13], antibacterial, antifungal [14] drugs, drugs for inhibitors [15] for growth of plant, curing hypoglycemia [16]. The versatile application of thioxoimidazolidine-4,5-dione make them paramount and privileged compounds. If carbonyl group of hydantoins are replaced thiocarbonyl group, it gives rise to thiohydantoin which have extensive applications in hypolipidemic, anticarcinogenic, antituberculosis, anti-herpes, anti-HIV anti-HSV, *etc*. These thiohydantoins are found to be significant antiinfalmatory [17], antiulcer, antimicrobial agents. Diverse studies [18-20] have described the synthesis of various amino acids and have used them as intermediates for the synthesis of heterocyclics [21,22]. A number of schemes are described for the synthesis of 2-thioxodihydro-1*H*-imidazole-4,5-dione derivatives [23-26] under solvent free conditions [27]. In the present paper, a safe and environmental friendly synthesis of 2-thioxoimidazolidine-4,5-dione using benzyl trimethylammonium hydroxide (Triton-B) as a phase transfer catalyst is discussed. We aim to overcome, the problems associated with other methods like higher reaction temperatures, longer reaction time, tedious work-up, lesser yields, *etc*.

EXPERIMENTAL

The reagents used in this research were procured from Merck, India. Products were ascertained by similarity in spectral data with the available compounds. NMR spectra were

obtained onBruker Advance spectrophotometer (400.13 MHz) with $CDCl_3$ as solvent and tetramethylsilane as reference. The elements in the compounds were confirmed by high resolution mass spectroscopy (HRMS) and it agreed approximately with the calculated mass obtained by HRMS. Structure of all products was drawn on similarity basis of the physical data with the reported data.

General procedure for the synthesis of compounds (1-15) using Triton-B: At room temperature, a solution of amine (1.0 mmol), CS_2 (10 mmol) and a Triton-B (40 wt. % in H_2O) (1.5 mmol) was stirred for 30 min, followed by addition of NH_4OH solution (28% NH_3 in H_2O) (1.1 mmol). The solution is again stirred at room temperature for 14 h. After stirring, Triton-B (40 wt. % in H_2O) (2.5 mmol) was added and again stirred for 15 more minutes. Then oxalylchloride (1 mmol) was added at room temperature. The reaction mixture was than stirred for 2 h. The progress of reaction was monitored by TLC. After completion, water (50 mL) was added and the product was extracted with ethyl acetate (3 × 20 mL). The combined organic layer was washed with brine, dried over sodium sulfate and concentrated under reduced pressure to afford crude product. This crude product was further purified by silica gel column chromatography by using eluent ethyl acetate:hexane (1:5) to afford pure product.

Spectral data

1-(4-(Trifluoromethyl)benzyl)-2-thioxoimidazolidine-4,5-dione (1): Yellow solid, m.p. = 232-234 ºC. Elemental analysis of $C_{11}H_7F_3N_2O_2S$ calcd. (found) %: C, 45.84 (46.14); H, 2.45 (2.72); F, 19.77 (20.17); N, 9.72 (10.32); O, 11.10 (12.10); S, 11.12 (12.12). ^{1}H NMR 400 MHz ($CDCl_3$): δ 9.296 (bs, 1H), 7.597 (d, *J* = 8.4 Hz, 2H), 7.525 (d, *J* = 8.0 Hz, 2H), 5.147 (s, 2H). ^{13}C NMR 400 MHz ($CDCl_3$): δ 44.475, 125.280, 125.929, 125.967, 126.004, 126.040, 129.497, 137.930, 156.141, 156.313, 177.846. MS (ESI): *m/z* $(M)^-$ calculated for $C_{11}H_7N_2O_2SF_3$: 288.02, Found $(M-1)^-$: 287.10.

1-(3-Chlorobenzyl)-2-thioxoimidazolidine-4,5-dione (2): Yellow solid, m.p. = 235-239 ºC. Elemental analysis of $C_{10}H_7N_2O_2SCl$ calcd. (found) %: C, 47.16 (48.12); H, 2.77 (3.17); Cl, 13.92 (14.82); N, 11.00 (12.07); O, 12.56 (13.46); S, 12.59 (13.49). ^{1}H NMR 400 MHz (DMSO-d_6): δ 13.104 (bs, 1H), 7.470 (s, 1H), 7.370-7.307 (m, 3H), 4.955 (s, 2H). ^{13}C NMR 400 MHz (DMSO-d_6): δ 43.187, 126.178, 127.246, 127.348, 130.122, 133.099, 137.804 156.383, 157.488, 182.763. MS (ESI): *m/z* $(M)^-$ calculated for $C_{10}H_7N_2O_2SCl$: 253.99, Found $(M-1)^-$: 253.01.

1-(4-(Trifluoromethyl)phenyl)-2-thioxoimidazolidine-4,5-dione (3): Yellow solid, m.p. = 231-234 ºC. Elemental analysis of $C_{10}H_5N_2O_2SF_3$ calcd. (found) %: C, 43.80 (44.42); H, 1.84 (2.14); F, 20.78 (21.38); N, 10.22 (11.12); O, 11.67 (12.47); S, 11.69 (12.60). ^{1}H NMR 400 MHz (DMSO-d_6): δ 13.405 (bs, 1H), 7.936 (d, *J* = 8.4 Hz, 2H), 7.609 (d, *J* = 8.4 Hz, 2H). ^{13}C NMR 400 MHz (DMSO-d_6): δ 125.216, 126.162, 126.234, 129.313, 129.632, 136.037, 155.667, 157.143, 182.003. MS (ESI): *m/z* $(M)^-$ calculated for $C_{10}H_5N_2O_2SF_3$: 274.0, Found $(M-1)^-$: 272.96.

1-(3-Chlorophenyl)-2-thioxoimidazolidine-4,5-dione (4): Yellow solid, m.p. = 233-237 ºC. Elemental analysis of $C_9H_5N_2O_2SCl$ calcd. (found) %: C, 44.92 (45.82); H, 2.09 (2.17); Cl, 14.73 (15.99); N, 11.64 (13.94); O, 13.30 (15.65); S, 13.32 (15.92). ^{1}H NMR 400 MHz (DMSO-d_6): δ 13.344 (bs, 1H), 7.608 (d, *J* = 8.4 Hz, 2H), 7.379 (d, *J* = 8.8 Hz, 2H). ^{13}C NMR 400 MHz (DMSO-d_6): δ 129.124, 130.364, 131.274, 133.866, 155.777, 157.223, 182.290.MS (ESI): *m/z* $(M)^-$ calculated for $C_9H_5N_2O_2SCl$: 239.98, Found $(M-1)^-$: 239.10.

1-Methyl-2-thioxoimidazolidine-4,5-dione (5): Yellow solid, m.p. = 225-229 ºC. Elemental analysis of $C_4H_4N_2O_2S$ calcd. (found) %: C, 33.33 (34.13); H, 2.80 (2.40); N, 19.43 (20.23); O, 22.20 (23.12); S, 22.24 (23.14). ^{1}H NMR 400 MHz ($CDCl_3$): δ 8.949 (bs, 1H), 3.406 (s, 3H). MS (ESI): *m/z* $(M)^-$ calculated for $C_4H_4N_2O_2S$: 144.0, Found $(M-1)^-$: 142.89.

1-(4-Fluorophenyl)-2-thioxoimidazolidine-4,5-dione (6): Yellow solid, m.p. = 230-235 ºC. Elemental analysis of $C_9H_5N_2O_2SF$ calcd. (found) %: C, 48.21 (48.81); H, 2.25 (2.75); F, 8.47 (8.97); N, 12.49 (12.99); O, 14.27 (14.57); S, 14.30 (14.93). ^{1}H NMR 400 MHz (DMSO-d_6): δ 13.328 (bs, 1H), 7.426-7.343 (m, 4H). ^{13}C NMR 400 MHz (DMSO-d_6): δ 115.916, 116.150, 128.641, 130.756, 130847, 157.319, 160.871, 163.319, 182.588. MS (ESI): *m/z* $(M)^-$ calculated for $C_9H_5N_2O_2SF$: 224.01, Found $(M-1)^-$: 223.08.

1-Phenyl-2-thioxoimidazolidine-4,5-dione (7): Yellow solid, m.p. = 232-236 ºC. Elemental analysis of $C_9H_6N_2O_2S$ calcd. (found) %: C, 52.42 (52.92); H, 2.93 (3.23); N, 13.58 (14.98); O, 15.52 (16.12); S, 15.55 (16.15). ^{1}H NMR 400 MHz ($CDCl_3$): δ 9.029 (bs, 1H), 7.7.536 (d, *J* = 6.8 Hz, 3H), 7.330 (d, *J* = 6.0 Hz, 2H). ^{13}C NMR 400 MHz ($CDCl_3$): δ 127.939, 129.670, 130.208, 131.200, 154.641, 177.688. MS (ESI): *m/z* $(M)^-$ calculated for $C_9H_6N_2O_2S$: 206.01, Found $(M-1)^-$: 205.12.

1-(3-Fluorophenyl)-2-thioxoimidazolidine-4,5-dione (8): Yellow solid, m.p. = 236-239 ºC. Elemental analysis of $C_9H_5N_2O_2SF$ calcd. (found) %: C, 48.21 (49.22); H, 2.25 (2.75); F, 8.47 (9.17); N, 12.49 (13.99); O, 14.27 (15.97); S, 14.30 (15.45). ^{1}H NMR 400 MHz (DMSO-d_6): δ 13.377 (bs, 1H), 7.586 (q, *J* = 6.8 Hz, 1H), 7.373 (t, *J* = 7.2 Hz, 1H), 7.230 (d, *J* = 8.0 Hz, 2H). MS (ESI): *m/z* $(M)^-$ calculated for $C_9H_5N_2O_2SF$: 224.01, Found $(M-1)^-$: 223.10.

1-(3,5-Dichlorophenyl)-2-thioxoimidazolidine-4,5-dione (9): Yellow solid, m.p. = 238-242 ºC. Elemental analysis of $C_9H_4N_2O_2SCl_2$ calcd. (found) %: C, 39.29 (40.21); H, 1.47 (2.17); Cl, 25.77 (35.17); N, 10.18 (11.78); O, 11.63 (12.33); S, 11.66 (12.96). ^{1}H NMR 400 MHz (DMSO-d_6): δ 13.515 (bs, 1H), 7.824 (s, 1H), 7.463 (s, 2H). ^{13}C NMR 400 MHz (DMSO-d_6): δ 127.642, 129.297, 134.116, 134.525, 156.428, 157.023, 181.706. MS (ESI): *m/z* $(M)^-$ calculated for $C_9H_4N_2O_2SCl_2$: 273.94, Found $(M-1)^-$: 272.94.

1-(4-Phenylbutyl)-2-thioxoimidazolidine-4,5-dione (10): Yellow solid, m.p. = 223-226 ºC. Elemental analysis of $C_{13}H_{14}N_2O_2S$ calcd. (found) %: C, 59.52 (60.12); H, 5.38 (6.18); N, 10.68 (11.18); O, 12.20 (13.21); S, 12.22 (13.92). ^{1}H NMR 400 MHz (DMSO-d_6): δ 13.002 (bs, 1H), 7.280-7.243 (m, 2H), 7.196-7.140 (m, 3H), 3.760 (t, *J* = 6.4 Hz, 2H), 2.578 (t, *J* = 6.8 Hz, 2H), 1.607-1.590(m, 4H). ^{13}C NMR 400 MHz (DMSO-d_6): δ 26.806, 28.022, 34.700, 40.383, 125.683, 127.246, 127.348, 128.205 128.305, 141.870, 156.215, s157.477, 182.941. MS (ESI): *m/z* $(M)^-$ calculated for $C_{13}H_{14}N_2O_2S$: 262.08, Found $(M-1)^-$: 261.11.

1-(4-Chlorophenyl)-2-thioxoimidazolidine-4,5-dione (11): Yellow solid, m.p. = 233-237 ºC. Elemental analysis of $C_9H_5N_2O_2SCl$ calcd. (found) %: C, 44.92 (45.62); H, 2.09 (2.17); Cl, 14.73 (15.93); N, 11.64 (12.93); O, 13.30 (13.98); S, 13.32 (14.95). ^{1}H NMR 400 MHz (DMSO-d_6): δ 13.344 (bs, 1H), 7.608 (d, J = 8.4 Hz, 2H), 7.379 (d, J = 8.8 Hz, 2H). ^{13}C NMR 400 MHz (DMSO-d_6): δ 129.124, 130.364, 131.274, 133.866, 155.777, 157.223, 182.290. MS (ESI): *m/z* $(M)^-$ calculated for $C_9H_5N_2O_2SCl$: 239.98, Found $(M-1)^-$: 239.10.

1-Ethyl-2-thioxoimidazolidine-4,5-dione (12): Yellow solid, m.p. = 224-230 ºC. Elemental analysis of $C_5H_5N_2O_2S$ calcd. (found) %: C, 37.97 (38.56); H, 3.82 (3.42); N, 17.71 (18.21); O, 20.23 (21.17); S, 20.27 (21.21). ^{1}H NMR 400 MHz ($CDCl_3$): δ 9.038 (bs, 1H), 4.007 (q, J = 7.2 Hz, 2H), 1.296 (t, J = 7.2 Hz, 3H). MS (ESI): *m/z* $(M)^-$ calculated for $C_5H_5N_2O_2S$: 158.01, Found $(M-1)^-$: 156.94.

1-(3-(Trifluoromethyl)phenyl)-2-thioxoimidazolidine-4,5-dione (13): Yellow solid, m.p. = 237-242 ºC. Elemental analysis of $C_{10}H_5N_2O_2SF_3$ calcd. (found) %: C, 43.80 (44.56); H, 1.84 (2.18); F, 20.78 (21.98); N, 10.22 (11.52); O, 11.67 (13.97); S, 11.69 (13.99). ^{1}H NMR 400 MHz ($CDCl_3$): δ 9.136 (bs, 1H), 7.870 (d, J = 7.2 Hz, 1H), 7.778-7.689 (m, 2H), 7.349 (d, J = 7.6 Hz, 1H). MS (ESI): *m/z* $(M)^-$ calculated for $C_{10}H_5N_2O_2SF_3$: 274.0, Found $(M-1)^-$: 273.07.

1-(4-Methylpentan-2-yl)-2-thioxoimidazolidine-4,5-dione (14): Yellow solid, m.p. = 225-230 ºC. Elemental analysis of $C_9H_{14}N_2O_2S$ calcd. (found) %: C, 50.45 (51.55); H, 6.59 (6.79); N, 13.07 (14.27); O, 14.93 (15.99); S, 14.96 (16.16). ^{1}H NMR 400 MHz ($CDCl_3$): δ 9.490 (bs, 1H), 4.927-4.871 (m, 1H), 3.900 (s, 1H), 2.116-2.039 (m, 1H), 1.554-1.452 (m, 1H), 1.419 (s, 3H), 0.905 (d, J = 6.4 Hz, 6H). MS (ESI): *m/z* $(M)^-$ calculated for $C_9H_{14}N_2O_2S$: 214.08, Found $(M-1)^-$: 213.09.

1-Phenethyl-2-thioxoimidazolidine-4,5-dione (15): Yellow solid, m.p. = 228-232 ºC. Elemental analysis of $C_{11}H_{10}N_2O_2S$ calcd. (found) %: C, 56.39 (57.34); H, 4.30 (4.51); N, 11.96 (13.26); O, 13.66 (15.96); S, 13.69 (15.99). ^{1}H NMR 400 MHz ($CDCl_3$): δ 9.413 (bs, 1H), 7.466 (d, J = 7.2 Hz, 1H), 7.375-7.27 (m, 4H), 3.726 (m, 2H), 1963 (m, 2H). ^{13}C NMR 400 MHz ($CDCl_3$): δ 43.187, 53.822, 126.393, 127.793, 127.951, 128.563, 128.967, 138.108, 154.782, 159.091, 177.068. MS (ESI): *m/z* $(M)^-$ calculated for $C_{11}H_{10}N_2O_2S$: 234.05, Found $(M-1)^-$: 233.05.

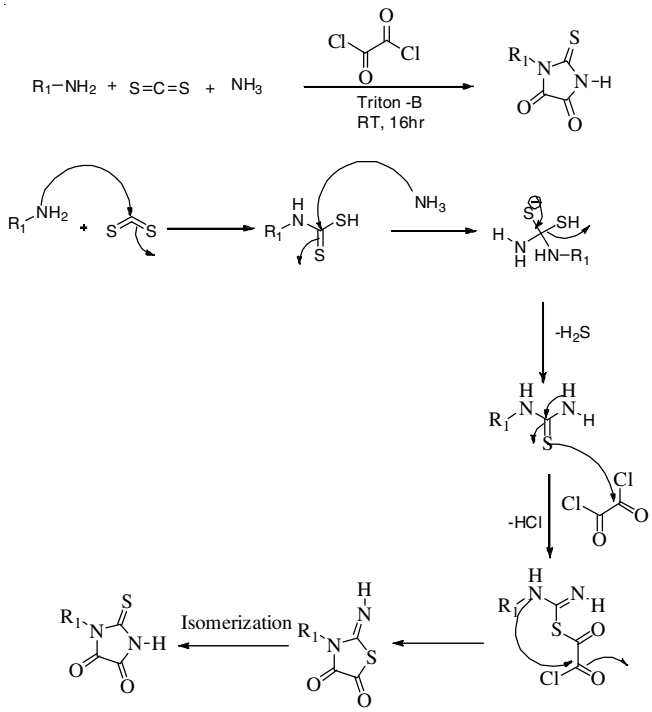
RESULTS AND DISCUSSION

In this communication, one-pot synthesis of 2-thioxoimidazolidin-4,5-diones (**1-15**) have been reported using Triton-B as phase transfer catalyst. The reaction between amine, CS_2, ammonia and oxalyl dichloride was spontaneous in the presence of phase transfer catalyst, Triton-B was completed with excellent yield as given in Table-1. The synthesized compounds are found to be soluble in acetone, methanol, ethanol, ethyl acetate, diethylether, dichlromethane, DMF, $CDCl_3$, *etc*. The confirmation of product formation was done with the aid of spectroscopic and analytical data. A number of phase transfer catalyst is frequently used for the synthesis [28] but Triton-B is preferable to other phase transfer catalyst due to high yields of the product [29]. The method is easier and require simpler work-up and low require cost less toxic reagent. Output of the product is improved and required less time for synthesis. The benefit of using Triton-B as phase transfer catalyst visit is recovered from the reaction mixture by filtration [30,31].

TABLE-1

Compd. No.	Substitution R_1	m.w.	Yield (%)
1	4-CF_3-C_6H_4-CH_2	288.02	95.01
2	3-Cl-C_6H_4-CH_2	253.99	94.53
3	4-CF_3-C_6H_4	274.22	92.53
4	3-Cl-C_6H_4	240.67	93.12
5	CH_3	144.15	94.35
6	4-F-C_6H_4	224.21	93.54
7	C_6H_5	206.22	90.12
8	3-F-C_6H_4	224.21	92.36
9	3,5-Cl_2-C_6H_3	275.11	93.87
10	C_6H_5-$(CH_2)_4$-	262.33	92.58
11	4-Cl-C_6H_5	240.67	93.25
12	C_2H_5	158.18	90.53
13	3-CF_3-C_6H_4	274.22	94.02
14	$(CH_3)_2$CH-CH_2-(CH_3)CH	214.28	92.30
15	$C_6H_5CH_2CH_2$	234.27	91.58

The compounds synthesized in this series were identified by high resolution mass spectroscopy (HRMS), ^{1}H NMR and ^{13}C NMR spectra. The intermediate most probably results from addition of oxalyl chloride to substituted thiourea and subsequently cyclization of the intermediate took place. Further isomerization of the intermediate gave way to the formation of the product (**Scheme-I**).



Scheme-I: Synthesis of 2-thioxoimidazolidin-4,5-dione catalyzed by Triton-B

Conclusion

Synthesis of 2-thioxoimidazolidine 4,5-diones has been reported by highly efficient method by coupling of primary amine, ammonia and oxalyl chloride *via* Triton-B/CS_2 system.

This Triton-B mediated synthesis is more convenient and safer than all other reported procedures like simplicity, normal temperature, easier and simpler work-up, better output and low cost reagents. This synthetic route thereby offers a more convenient approach for formation of C-S bonds essential to numerous organic syntheses.

CONFLICT OF INTEREST

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

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