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# Ultrasound promoted greener synthesis of spiro[indole-3,5′-[1,3]oxathiolanes in water

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ABSTRACT

An aqueous mediated novel synthesis of substituted 2′amino-4′benzoyl-2′-methyl spiro[indole 3,5′-[1,3]oxathiolane]-2(1H)-ones (**2a–f**) was carried out from the reaction of spiro [indole-3,2′-oxiranes] (**1a–f**) with thioacetamide in the presence of LiBr as catalyst. The reaction was carried out under both microwaves and sonication and results were also compared with conventional method. In general, improvement in rate and yields observed when reaction was carried out under sonication as compared to microwave irradiation and conventional method.



## 1. Introduction

Heterocyclic rings have played an important role in medicinal chemistry, serving as key template central to the development of numerous important therapeutic agents. Among the many five-membered heterocycles studied, 1,3-oxathiolanes are one such class of heterocycles which attracted much attention as they have been reported to possess a wide range of biological activities including anti-viral [1], anticonvulsant [2], antiulcer [3] and anti-fungal activity [4]. In addition they also showed antihepatitis B virus, anti-HIV and anti-HBV activity [5]. 1,3-oxathiolane derivatives are novel precursors of 2′,3-dideoxy-3′-oxa-4′′thioribonucleosides which also show anti-viral activities [6]. Spirocyclic indolines represent important scaffolds for drug discovery [7]. These heterocycles offer well-defined substituent vectors and their conformational rigidity, small size, and polarity confers favorable physical properties for oral bioavailability. On the other hand oxindole derivatives have been identified as a novel inhibitor of microtubule assembly [8], utilized as glycine receptor [9], nonpeptidyl growth-hormones secretagogues [10], muscarinic receptor modulators [11] and identified as privileged scaffold for the discovery of ligands for G-protein coupled receptor [12].

The common methods used for the preparation of 1,3-oxathiolanes include the reaction of aromatic thioketone with monosubstituted oxirane [13], xanthene thione and by the reaction of 4,4′-dimethoxythiobenzophenone, xanthene thione and admantane-2-thione with 2-vinyloxirane [14], by condensation of glyoxylic acid ester with 2,5-dihydroxy-1,4-dithianes [15], by the reaction of 1,3-thiazole-5(4H)-thiones with 1,2-epoxy cycloalkanes [16] and the reaction of epoxides with sulfur and carbon monoxide [17], by the reaction of benzoyloxy acetaldehyde and p-dithiane-2,5-diol [18], by the reaction of oxirane and carbon disulphide [19] etc.

Many of these methods suffer from several limitations such as, longer reaction time, unsatisfactory yield, harsh reaction condition and excessive use of reagents and catalyst. In order to over came these limitations and in vision of the requirement of green chemistry it is therefore important to find convenient method for the preparation of these compounds.

In the last few years the development of synthetic protocols employing ultrasound irradiation has determined an epoch-making change in organic reactions and activates poorly reactive substrates [20]. The notable features of the ultrasound approach are enhanced reaction rates, formation of purer products in high yields, easier manipulation and considered as processing aid in terms of energy conservation and waste minimization compared with traditional methods [21]. Further, among alternatives, water is very begin solvent for organic transformation offers, green chemistry benefits and expedite the synthesis of diverse heterocycles [22].

As a part of our interest in the synthesis of a wide range of heterocyclic systems, and in a continuation of using green chemistry tools for heterocyclic synthesis [23], we performed the novel synthesis of spiro[indole-oxathiolanes] (**2a–f**) by the reaction of

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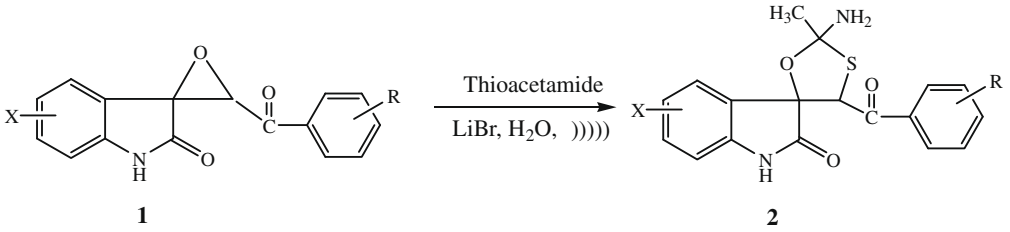



**Scheme 1.**

spiro[indole-oxiranes] (**1a–f**) with thioacetamide in presence of LiBr under ultrasound irradiation and employing water as the reaction medium for the first time. In fact, as clearly stated by Sheldon, it is generally recognized that "The best solvent is no solvent and if a solvent (diluent) is needed it should preferably be water [24]". To the best of our knowledge no report is available in the literature using ultrasonic assisted method for this transformation (Scheme 1).

## 2. Results and discussion

The required precursors spiro[indole-3,2′-oxirane]3′ benzoyl-2(*1H*)-ones (**1a–f**) were synthesized by our improved method involving the reaction of 3-aroylmethyllene indol-2-one with alkaline $H_2O_2$ under microwave irradiation in 2–3 min at 240 W [25].

To synthesize spiro[indole-1,3-oxathiolane] (**2a**), the reaction of **1a** with the thioacetamide in presence of LiBr has been carried out both under low intensity ultrasonic (LIU) laboratory cleaning bath (which is more economic) and high intensity ultrasound probe system (HIU) for comparative studies [26]. Although, it is well established that LIU from an ultrasonic cleaner has considerably less power and the energy of ultrasound is not uniformly available from the ultrasonic bath [27] when compared to HIU from a direct immersion horn [28]. This can lead to reproducibility problems due to the lower power involved for LIU [29]. In the present case, reaction was facile using both instruments but excellent yield in shorter reaction time with reproducible results was achieved using (HIU) sonicator as compared to (LIU) ultrasonic bath. Hence, other compounds listed in Table 2 were synthesized under HIU irradiations.

**Table 1**
Comparative study for synthesis of spiro[indole-1,3-oxathiolane] **2a**.

| S. no. | Reaction condition | Method | Time (min/h) | Yield (%) |
|---|---|---|---|---|
| 1 | Water | US bath (LIU) | 50 min | 80 |
| 2 | Water | Sonicator (HIU) | 7 min | 84 |
| 3 | Water | MW | 20 min | 76 |
| 4 | DMF | MW | 15 min | 70 |
| 5 | THF | Stirring | 5 h | 50 |
| 6 | Water | Stirring | 5 h | 37 |

It appears that in the present heterogeneous system sonochemical activation is mainly a consequence of the mechanical effects of cavitation [30], a liquid jet propagates towards the phase boundary at a velocity of several hundred meters per second, and hits the surface violently. At a liquid–liquid interface, the mutual injection of droplets results in emulsification. On a solid, the intense physical stresses produce particle breakage, the importance of which depends, *inter alia*, on the lattice energy of the solid [31]. In addition, cavitation greatly accelerates mass transport [32] and repassivation by reaction products is made less important or even avoided [33]. Further, there are certain cases being reported, in which sonication induces a specific reactivity under both homogeneous and heterogeneous conditions [34]. Since purely mechanical effects cannot explain such qualitative changes, cavitation must have direct chemical consequences.

In continuation of our earlier interest on microwave-assisted reactions we have also carried out the synthesis of **2a** under microwave irradiation using water/DMF as a solvent (Table 1). The role of DMF can be explained as energy transfer agent and homogenizer to increase the reaction temperature [35]. There was no appreciable increase in yield, thus the effect of microwaves on the synthesis of spiro derivative in not as efficient as ultrasound, but still better than the thermal method in which the mixture of products was formed.

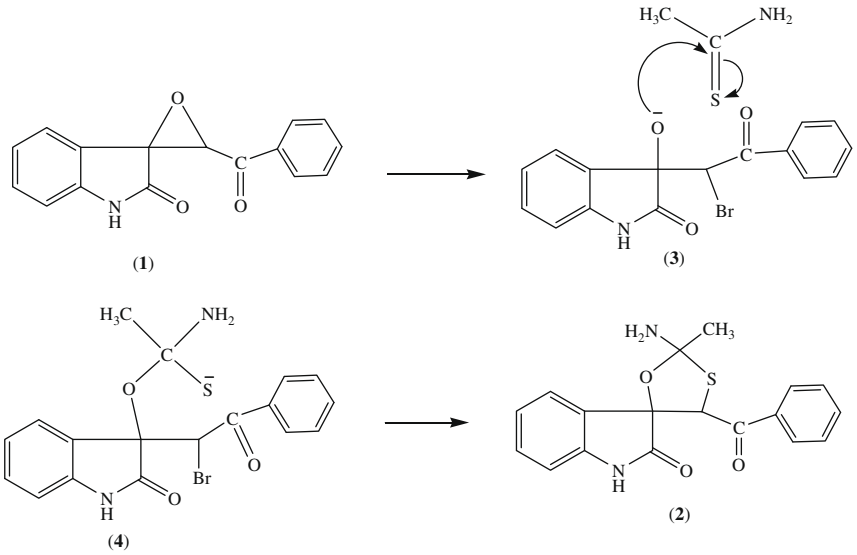
Hence, a series of spiro[indole1,3-oxathiolanes] (**2a–f**) was synthesized under sonication using water as solvent. To find the specific effect of ultrasound on this (HIU) the reaction was also carried out under same conditions in absence of ultrasound irradiation (Table 1). It was observed that the reaction time increased considerably and the yield of the product decreased due to formation of mixture of products. Thus, ultrasound irradiation was found to have beneficial effect on the synthesis of spiro [indole-oxathiolanes].

The plausible mechanism for present investigation involves nucleophilic attack of bromide ion at less substituted C-3 position of the spiro epoxide [36] leading to the formation of intermediate (3 and 4). Further, sulfur being a better nucleophile than nitrogen atom so in the next step cyclization leads to formation of oxathiolane ring [37] (Scheme 2).

The structure of the all synthesized compounds (**2a–f**) was established by their spectral and analytical studies.

**Table 2**
Synthesis of 2′amino-4′benzoyl-2′-methyl spiro[indole-3,5′-[1,3]oxathiolane]-2(1H)-ones (**2a–f**) under sonication and conventional conditions.

| Compound | X | R | Ultrasonic irradiation | | Conventional | | M.P. (°C) |
|---|---|---|---|---|---|---|---|
| | | | Time (min) | Yield (%) | Time (h) | Yield (%) | |
| **2a** | H | H | 7 | 84 | 5 | 60 | 235 [38] |
| **2b** | 5-Cl | H | 6 | 87 | 5 | 64 | 265 |
| **2c** | H | 4-F | 5 | 86 | 5 | 58 | 225 |
| **2d** | 5-Br | H | 6 | 87 | 5 | 61 | 250 |
| **2e** | 5-F | 4-Cl | 7 | 82 | 5 | 59 | 240 |
| **2f** | 5-$CH_3$ | 3-Cl | 8 | 86 | 5 | 62 | 270 |



**Scheme 2.**

## 3. Conclusion

In conclusion we have developed an effective methodology for the synthesis of spiro derivatives. This new methodology offers several advantages such as simple procedure, low cost, easy work-up, short reaction times and milder conditions. In addition this series may prove new classes of biological active compound for biomedical screening, which is in progress.

## 4. Experimental

Melting points were determined on a Toshniwal apparatus. The purity of compounds was checked on thin layers of silica gel in various non-aqueous solvent systems, for e.g. benzene:ethylacetate (9:1), benzene:dichloromethane (8:2). IR spectra (KBr) were recorded on a Magna FT IR–550 spectrophotometer and $^1$H NMR and $^{13}$C NMR spectra were recorded on Bruker DRX-300 using $CDCl_3$ at 300.15 and 75.47, respectively. TMS was used as internal reference. Mass spectrum of representative compound was recorded on Kratos 50 mass spectrometer at 70 eV. The microwave-assisted reactions were carried out in a multimode MW oven (Panasonic-NN-781JF) operating at 1000 W generating 2450 MHz frequency and ultrasonic bath (Bandelin Sonorex) operating at 230 V generating 33 kHz output frequency. HIU irradiation was provided by an ultrasonic processor probe system (Processor SONOPROS PR-1000MP, OSCAR ULTRASONICS made) operating at 20 kHz, 750 W with 6 mm/12 mm tip diameter probes.

### *4.1. Synthesis of 2′amino-4′benzoyl-2′-methyl spiro[indole-3,5′-[1,3]oxathiolane]-2(1H)-ones (**2a–f**)*

#### *4.1.1. Conventional synthesis*

A mixture of spiro[indole-3,2′-oxirane]3′-benzoyl-2(1H)-ones (2 mmol), LiBr (.2 mmol) in 10 ml THF was stirred at room temperature for 5 min, then thioacetamide (2 mmol) was added to the solution and resulting mixture was further stirred at room temperature for 5 h. Progress of the reaction was monitored by TLC. After completion of reaction, the solvent was removed under reduced pressure and the residue was purified on a silica gel column to give **2a**.

#### *4.1.2. Reaction under ultrasound irradiation*

(i) *Reaction under HIU irradiation using sonicator:* A mixture of spiro[indole-3,2′-oxiran]3′-benzoyl-2(1H)-one (**1a)** (2 mmol), LiBr (.2 mmol) and thioacetamide (2 mmol) in 10 ml water were taken in a flask. The flask was attached to a 12 mm tip diameter probe and the reaction mixture was sonicated for the specified period at 50% power of the processor and 230 W output in a 4 s pulse mode. At the end of the reaction period, TLC was checked and the flask was detached from the probe and the content was transferred into a beaker. The formed solid was filtered off, washed thoroughly with water to obtain the pure compound **2a**.

(ii) *Reaction under LIU irradiation using ultrasound bath:* A mixture of spiro compound (**1a)** (2 mmol), LiBr (.2 mmol) and thioacetamide (2 mmol) in 10 ml water were taken in a conical flask, which was immersed in water bath of an ultrasonic cleaner. The flask was positioned 0.5 cm above the bottom of the bath at room temperature until the completion of the reaction (monitored by TLC). The formed solid was filtered off, washed with water to afford the spiro compound **2a**.

#### *4.1.3. Microwave mediated synthesis*

(i) *Using DMF:* A mixture of spiro compound (**1a)** (2 mmol), LiBr (.2 mmol) and thioacetamide (2 mmol) with few drops of DMF contained in a beaker was placed in the microwave oven and irradiated for appropriate time (Table 1) The reaction mixture was cooled and poured onto crushed ice. The precipitate thus filtered, washed with water and recrystallized from ethanol.

(ii**)** *Using water*: A mixture of spiro compound (**1a)** (2 mmol), LiBr (.2 mmol) thioacetamide (2 mmol) and water as a solvent in open borosil beaker was irradiated inside microwave oven at 640 W till the completion of reaction (TLC). An oily product was formed which was solidified on standing, washed with water and recrystallized from ethanol.

**Compound 2a**: IR (KBr, $cm^{-1}$) $V_{max\ 3250–3460}$ (NH and $NH_2$) 1715 (C=O), 1695 (C=O); $^1$H NMR (300 MHz, $CDCl_3$) $\delta_H$: 2.24 (s, 3H, $CH_3$), 5.12 (s, 1H, CH), 6.23–7.87 (m, 11H, Ar–H, $NH_2$), 8.15 (bs, IH, NH); $^{13}$C NMR (74.46 MHz, $CDCl_3$) $\delta_C$: 32.3 ($CH_3$). 61.7 (CH), 89.7 (spiro carbon), 114.21–142.3 (aromatic carbons), 177.3 (C=O), 199.8 (C=O), MS (*m/z*): 340.09 (100.0%), 341.09 (21.9%), 342.08 (4.4%), 342.09 (2.8%), Anal. calc. for $C_{18}H_{16}N_2O_3S$: C, 63.51; N, 8.23; S, 9.42; Found C, 63.21; N, 8.20; S, 9.40.

**Compound 2b**: IR (KBr, $cm^{-1}$) $V_{max}$ 3255–3470 (NH and $NH_2$) 1717 (C=O), 1690 (C=O); $^1H$ NMR (300 MHz, $CDCl_3$) $\delta_H$: 2.23 (s, 3H, $CH_3$), 5.14 (s, 1H, CH), 6.24–7.89 (m, 10H, Ar–H, $NH_2$), 8.18 (bs, IH, NH); $^{13}C$ NMR (74.46 MHz, $CDCl_3$) $\delta_C$: 31.9 ($CH_3$). 61.9 (CH), 90.1 (spiro carbon), 114.21–142.3 (aromatic carbons), 177.7(C=O), 197.8 (C=O), MS (*m/z*): 374.05 ($M^+$). Anal. calc. for: $C_{18}H_{15}ClN_2O_3S$: C, 57.68; H, 4.03; N, 7.47; S, 8.55. Found C, 57.88; H, 4.01; N, 7.45; S, 8.52.

**Compound 2c**: IR (KBr, $cm^{-1}$) $V_{max}$ 3257–3468 (NH and $NH_2$) 1716 (C=O), 1699 (C=O), $cm^{-1}$: $^1H$ NMR (300 MHz, CDCl3) $\delta_H$: 2.22 (s, 3H, $CH_3$), 5.15 (s, 1H, CH), 6.30–7.89 (m, 10H, Ar–H, $NH_2$), 8.16 (bs, IH, NH); $^{13}C$ NMR (74.46 MHz, $CDCl_3$) $\delta$ 33.3 ($CH_3$). 62.7 (CH), 89.2 (spiro carbon), 118.2–145.3 (aromatic carbon), 179.2 (C=O), 198.6 (C=O), MS (*m/z*): 358.08 (M). Anal. calc. for $C_{18}H_{15}FN_2O_3S$; C, 60.32; H, 4.22; N, 7.82; S, 8.95. Found C, 60.11; H, 4.24; N, 7.86; S, 8.91.

**Compound 2d**: IR (KBr, $cm^{-1}$) $V_{max}$ 3270–3480 (NH and $NH_2$) 1719 (C=O), 1698 (C=O), $cm^{-1}$: $^1H$ NMR (300 MHz, $CDCl_3$) $\delta_H$: 2.28 (s, 3H, $CH_3$), 5.10 (s, 1H, CH), 6.28–7.84 (m, 10H, Ar–H, $NH_2$), 8.18 (bs, IH, NH); $^{13}C$ NMR (74.46 MHz, $CDCl_3$) $\delta$ 34.3 ($CH_3$). 61.7 (CH), 85.7 (spiro carbon), 119.21–147.3 (aromatic carbon, 179.3 (C=O), 192.8 (C=O): Anal. calc. for $C_{18}H_{15}BrN_2O_3S$: C, 51.56; H, 3.61; N, 6.68; S, 7.65. Found C, 51.26; H, 3.60; N, 6.70; S, 7.61.

**Compound 2e**: IR (KBr, $cm^{-1}$) $V_{max}$ 3275–3465 (NH and $NH_2$) 1720 (C=O), 1700 (C=O) $cm^{-1}$: $^1H$ NMR (300 MHz, $CDCl_3$) $\delta_H$: 2.29 (s, 3H, $CH_3$), 5.15 (s, 1H, CH), 6.33–7.89 (m, 9H, Ar–H, $NH_2$), 8.15 (bs, IH, NH) $^{13}C$ NMR (74.46 MHz, $CDCl_3$) $\delta$ 34.3 ($CH_3$). 61.7 (CH), 89.1 (spiro carbon), 124.21–150.3 (aromatic carbon, 175.3 (C=O), 197.8 (C=O), MS (*m/z*): 392.04 (100.0%). Anal. calc. for $C_{18}H_{14}ClFN_2O_3S$: C, 55.03; H, 3.59; N, 7.13; S, 8.16. Found C, 55.23; H, 3.58; N, 7.15; S, 8.13.

**Compound 2f**: IR (KBr, $cm^{-1}$) $V_{max}$ 3280–3468 (NH and $NH_2$) 1717 (C=O), 1694 (C=O) $cm^{-1}$: $^1H$ NMR (300 MHz, CDCl3) $\delta_H$: 2.25 (s, 3H, $CH_3$), 2.27(s, 3H, $CH_3$), 5.17 (s, 1H, CH), 6.30–7.80 (m, 11H, Ar–H, $NH_2$), 8.13 (bs, IH, NH); $^{13}C$ NMR (74.46 MHz, $CDCl_3$) $\delta$ 32.0 ($CH_3$). 61.7 (CH), 89.2 (spiro carbon), 119.21–150.3 (aromatic carbon), 176.3 (C=O), 197.8 (C=O). Anal. calc. for $C_{19}H_{17}ClN_2O_3S$: C, 58.68; H, 4.41; N, 7.20; S, 8.25. Found C, 58.49; H, 4.42; N, 7.23; S, 8.22.

Presence and position of NH and $NH_2$ protons were confirmed by deuterium exchange.

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