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A short review: Chemistry of thioisatin and its derivatives

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In recent years, the thioisatin (benzo[b]thiophene-2,3-diones) have attracted considerable attention due to their ability to act as intermediates in the preparation of a series of fused spiroheterocycles. The benzothiophenic frameworks are important targets in synthetic and medicinal chemistry because this fragment is a key moiety in a wide number of natural and synthetic agents. Subsequently the present study deals with the synthesis of a variety of spiroheterocycles for which different strategies have been evolved *viz.* [4+2] photocycloaddition (spiropyrans), cyclocondensation (benzodiazocine), nucleophilic cyclization (spirothioazolidinones) and air oxidation (disulfides). semicarbazide reacts with thioisatin at 3-position (thiosemicarbazones, 2-hydroxy-(2*H*)-3,3-diaryl- and 3-hydroxy-3*H*-2,2-diaryl-benzo[b]thiophenes hydroxyl compounds), 4,9-dimethylthiophanthrenes.

Keywords: Thioisatin, reactivity of β-carbonyl functionality, disulfides derivatives, photocycloaddition reactions

The synthesis of privileged classes of heterocyclic compounds has become one of the prime areas of research in the field of synthetic and medicinal chemistry. Most of the compounds participate in important biochemical processes and are the constituents of main substances (DNA, RNA) in living cells. For more than a century, heterocyclic compounds have ranked amongest the most important compounds and the fused heterocyclic ring systems have emerged as the important scaffolds' in medicinal chemistry[1] and appear in several natural and synthetic compounds of significant pharmacological properties[2]. In recent years, the benzo[b]thiophene-2,3-diones have attracted considerable attention due to their ability to act as intermediates in the preparation of a series of fused benzothiazolidinones[3]. Benzothiophene frameworks is a core structure in the most popular commercial drugs[4] *i.e.* Sertaconazole (Dermofix), Raloxifine (Evista), Metizoline, *etc*. In Sertaconazole benzothiophene ring mimics tryptophan (that is found in the fungal membrane in addition to lipid) and increases the drugs ability to form the pores in the fungal cell membranes. Raloxifeine (Evista) is used in the prevention of osteoporosis in postmenopausal women[5]. Raloxifene caused fewer uterine cancers as was announced by "National Cancer Institute'' in 2006. Metizoline is used as a vasoconstrictus and nasal decongestant drug.

Amongst the various fused heterocycles, thiophene moieties fused with benzene ring *viz*. thioisatin or benzo[b]thiophene-2,3-diones (Figure 1) have important applications in synthetic organic chemistry[6-10].

For the preparation of thioisatin or benzo[b]thiophene-2,3-dione, thiophenol **(1a)** was reacted with oxalyl chloride in refluxing diethyl ether. This is followed by intramolecular Friedel Crafts cyclisation of the intermediate acyl chloride (**2**) with $AlCl_3$ to afford the benzo[b]thiophene-2,3-dione (thioisatin) (Scheme I) in 75% yield.

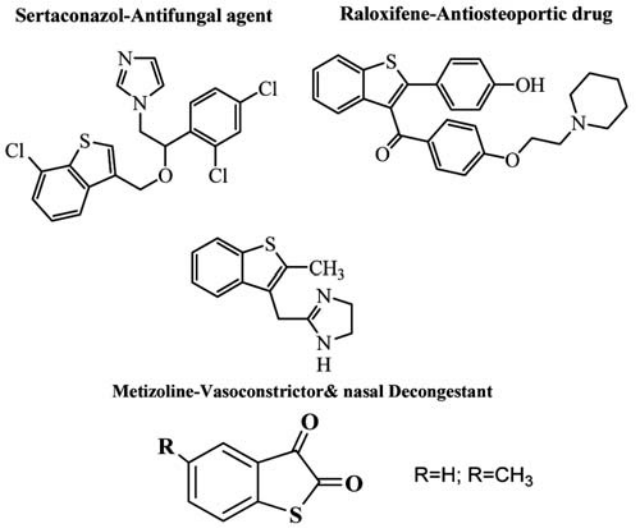


Figure 1 — Benzo[b]thiophene-2,3-diones

5-Methylbenzo[b]thiophene-2, 3-dione (5-methyl thioisatin) was also prepared by the similar procedure using 4-methylthiophenol and oxalyl chloride in 69% yield. The crude products were purified by column chromatography over silica gel wherein shiny reddish and orange crystals were obtained from petroleum ether-chloroform (4:1) as eluent fraction.

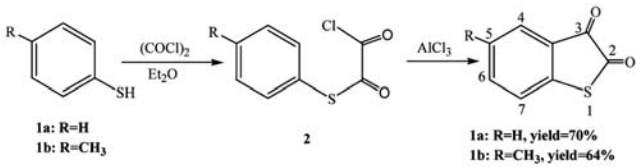
The IR spectrum of thioisatin **(1a)** showed absorption peak at 3100 for C-H aromatic, 2890 for C-H aliphatic, 1730 and 1710 for S–C=O and >C=O respectively and a peak at 675 cm^{-1} was assigned to C-S vibrations. In the ^1H NMR spectrum, signal for 5-H and 6-H resonated at δ 7.20-7.33 ppm as multiplet and a double doublet was observed for 7-H at δ 7.55 (J_1 = 7.0 Hz, J_2 = 1.5 Hz). A doublet was obtained for 4-H at δ 7.71 (J = 7.4 Hz).

In case of 5-methylthioisatin **(1b)** its IR spectrum showed following absorption peaks: 1734 and 1707 cm^{-1} ($\nu_{C=O}$), 690 cm^{-1} (ν_{C-S}) vibrations. In the ^1H NMR, methyl protons showed a singlet at δ 2.314 and a doublet was observed for 6-H at δ 7.42 (J = 8.1 Hz). The 4-H proton and 7-H protons resonated as doublets at δ 7.59 (J = 7.2 Hz) and δ 7.70 ppm (J = 6.2 Hz) respectively. In ^{13}C NMR spectrum the two carbonyl carbons resonated at δ 186.79 ppm (2-C=O) and δ 182.51 ppm (3-C=O) respectively. Other signals obtained are: aromatic carbons (C-7a) at δ 139.78, (C-4) at δ 139.14, (C-3a) at δ 138.46, (C-7) at δ 129.09, (C-6) at δ 125.63, (C-5) at δ 124.92 and methyl carbon resonated at δ 21.28 ppm. Its mass spectrum showed molecular ion peak (M^+) at m/z 177.9 (15%) which produced a base peak at m/z 149.9 [100%] by loss of CO moiety as depicted in Scheme II.

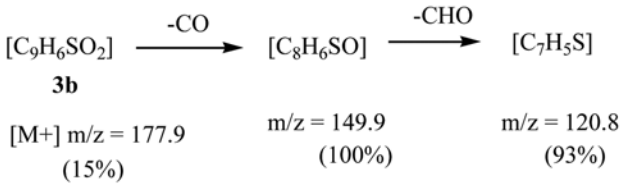
The C-3 carbonyl group of benzo[b]thiophene-2,3-dione (thioisatin) is strongly electrophilic[11]. Thioisatin, is briefly reported in condensation and addition reactions[12]. To widen the scope, we have synthesized novel compounds from thioisatin and its derivatives *viz*. spiropyrans, 8-membered benzodiazocines, thiazolidinones and diphenyldisulfides. In most of these reactions, chalcones act as intermediates and can inhibit glutathione-S-transferases (GSTs) enzymes that appear to be involved in drug resistance[13] and it also seems to be promising antifungal drug and may inhibit the growth of various fungi and yeast[14]. Spiropyran heterocyclics are of considerable interest because the conformational restriction associated with the structural rigidity influences considerably their biological activity[15]. Therefore the spiropyrans structural system is also a core skeleton of many bioactive natural products such as cytotoxic rotenoid amorphispironone, antimalarials, robustadials and antileishmanial euglobals[16,17]. Synthetic spirobenzopyranes, such as sorbinil and SNK-860, have been shown to be potent aldose reductase inhibitors[18] and hence reported synthesis may be potentially useful to prevent long-term diabetic complications. Several benzodiazocine derivatives have been shown to be pharmacologically useful such as antiviral, cholesterol-lowering and hormone-like activity[19-22]. Additionally some members of diazocine system have found applications as homologues of benzodiazepine drugs and as reversal agents in multidrug resistance[23-25]. In recent years material chemists have explored the electrochemical properties of diazocines, which were found to be useful as a basis for molecular machines and artificial muscles[26,27].

The disulfides have emerged as a valuable tool in biochemistry, medicinal and biological chemistry due to their selective formation in the presence of other functional groups and reversibility of their formation under reducing conditions[28]. Hence, disulfide bond formation has been used for facilitating protein refolding and for the synthesis of prodrugs with increased hydrophobicity or cell permeability[29]. Inside the cell the disulfide bond is then conveniently reduced as a result of the high glutathione levels, thus releasing the active compounds[30-32].

It is evident from the above discussion that synthesized scaffolds may play a crucial role in search for new pharmacological agents and drugs and hence a detailed methodology, application and reactivity of analogues of synthesized heterocyclic motif's is described in the following sections.



Scheme I



Scheme II — Mass fragmentation pattern of 5-methylthioisatin

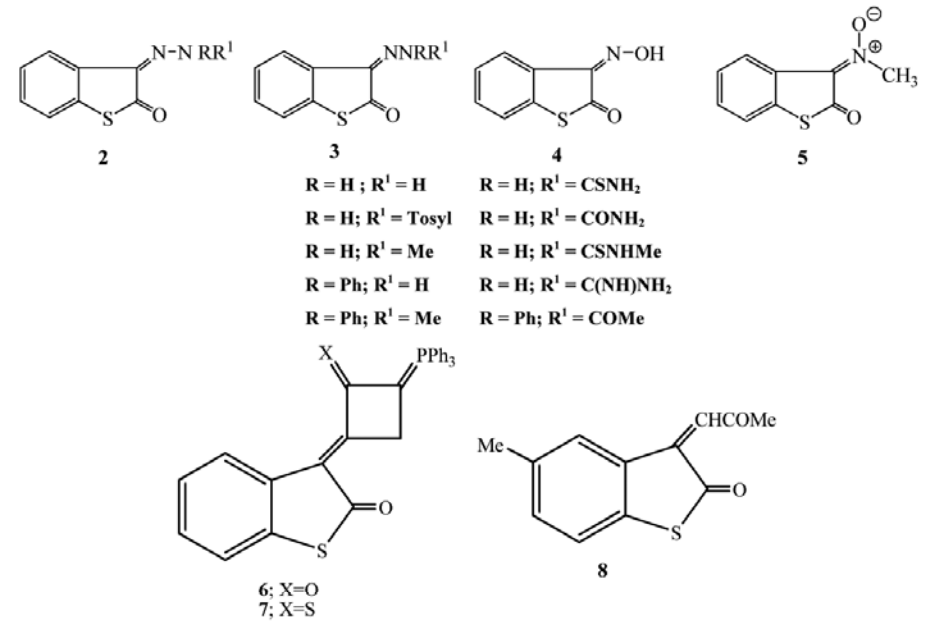
Synthesis and reactions of thioisatin derivatives: reactivity of β- carbonyl functionality

i. Thioisatin react with hydrazines to give hydrazones (**2**) with the condensation taking place at the β-carbonyl[33,34]. Similarly thiosemicarbazides and semicarbazides reacts with thioisatin at 3-position to give thiosemicarbazones (**3**). Hydroxlyamines[35] and N-methylhydroxylamines afford (**4**) and (**5**) respectively.

ii. A novel synthesis of phosphoanylidene-cyclobutane diones (**6**) and dithioxo cyclobutylidenes (**7**) from the reaction of Ph_3P+C- C-X with benzo[b]thiophene-2, 3-diones has been reported[36]. The reaction of stabilized 2-oxoalkylidene phosphoranes, eg., Ph_3P:CHCOMe, and unsaturated ketone (**8**) was also studied.

iii. The synthesis a few 2-hydroxy-(2*H*)-3, 3-diaryl- and 3-hydroxy-3H-2,2-diaryl-benzo[b]thiophenes hydroxyl compounds is described[37]. By acidic treatement these compounds readily rearrange to 2,3-diaryl-benzo[b]thiophenes in almost quantitative yields (Scheme III).

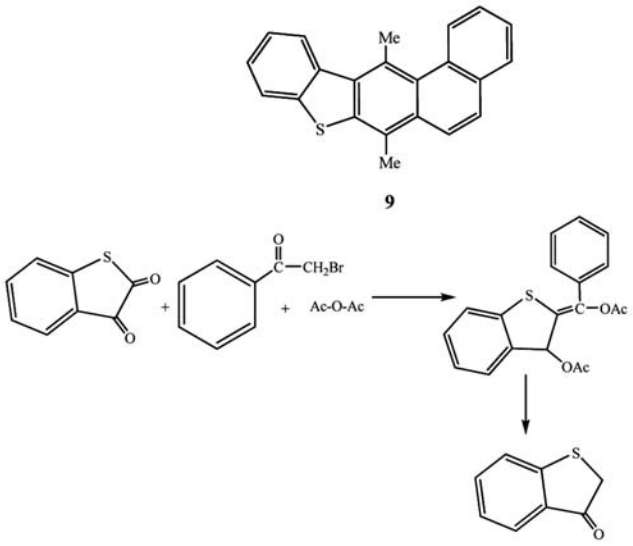
iv. Thioisatin was caused to react with ω-chloroacetophenone, 1-bromoacetyl- or 2-bromoacetylnaphthalene in alkaline solution followed by cyclization of the intermediate to give quinones. The quinones were then



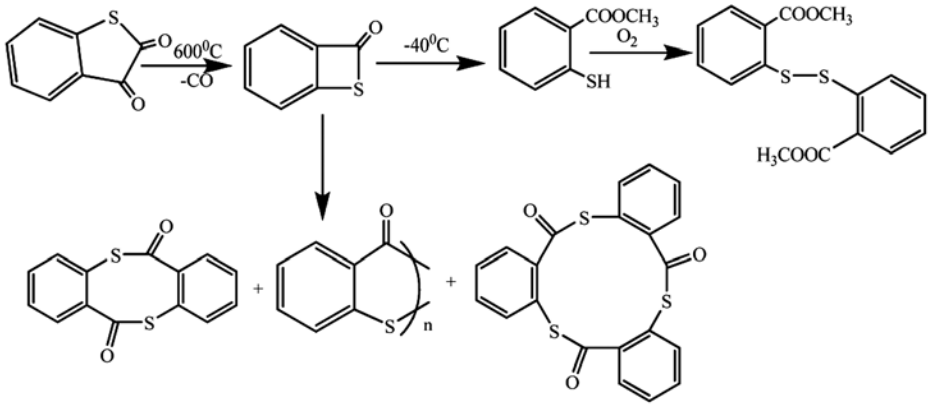
Scheme III

converted to the corresponding 4,9-dimethylthiophanthrenes (**9**)[38].

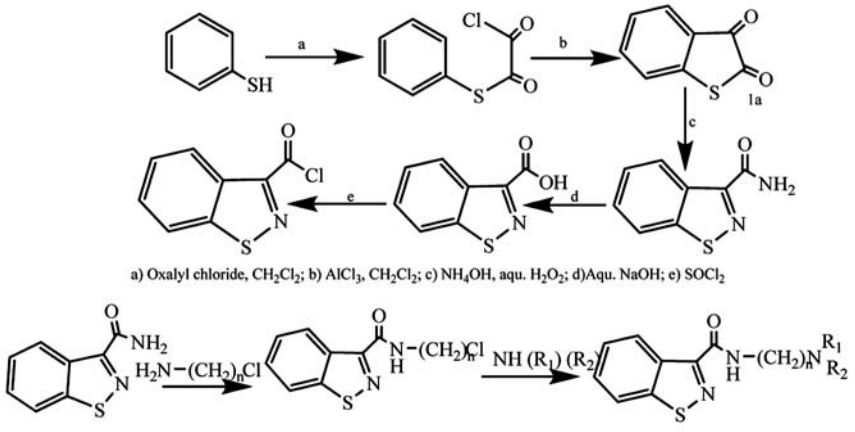
i. The use of 2,3-dihydrobenzo[b]thiophene-2, 3-dione as a source of 2-mercaptophenylacetic acid permits a convenient preparation of 2-carboxymethyl thiophenyl acetic acid or derivatives. These undergo a Dieckmann cyclization to 3-acetoxybenzothiopyrans which may be hydrolysed to thiochroman-3-ones (Scheme IV)[39].
ii. Benzothiet-2-one is obtained as a neat solid, stable below −20°C (−45°C in solution), by flash vaccum pyrolysis of benzothiophene-2,3-dione. The thietones undergo rapid ring opening reactions with methanol to give the corresponding carboxylic acid esters. Benzothietone dimerizes, trimerizes and polymerizes and the polymer is by far the major product (Scheme V)[40].
iii. A series of benzisothiazole-3-carboxamides have prepared and tested for potential antipsychotic activity. In general, the compounds showed an affinity for dopamine D_2 and serotonin 5 HT_{2A} and $5HT_{1A}$ receptors (Scheme VI)[41].
iv. Cyclic secondary α-amino acids react with isatin and methyl acrylate *via* decarboxylative



Scheme IV



Scheme V



Scheme VI

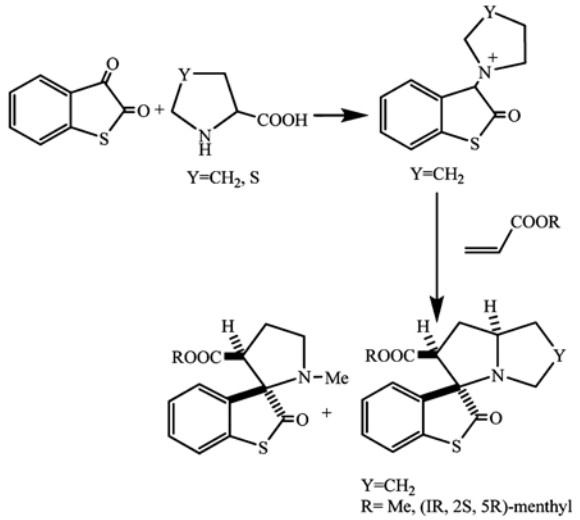
azomethine ylide formation and subsequent cycloaddition involving an *endo* transition state to give cycloadducts in 67-82% yield (Scheme VII)[42].

v. Thioisatin reacts with malononitrile in the presence of triethylamine with ring expansion to yield thiocoumarin. In the absence of triethylamine, the reaction yields condensation product without ring opening (Scheme VIII)[43].

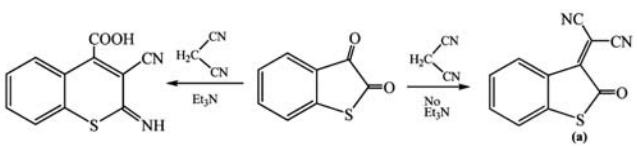
vi. 2,3-Thianaphthene-quinones were successively converted to thianahthene-2, 3-dicarboxylic acids, their imides, diamides and finally thianaphthene-2, 3-dicarbonitriles the key precursors for series of novel porphyrazines bearing four 2, 3-annulated thianaphthene moieties (Scheme IX)[44].

vii. Indigo and a number of indigoid dyes belonging to the indole or thionaphthene series are more or less readily decomposed by alkalies in *o*-H_2N-C_6H_4-COOH or HS-C_6H_4COOH and a cyclic *o*-hydroxy aldehyde (Scheme X).

viii. Verma *et al.*[45] have accomplished a simple and efficient method for the synthesis of novel spiropyrrolidines by regioselective 1,3-dipolar cycloaddition reactions of azomethine ylide generated by benzo[b]thiophene-2,3-dione and piperidine 2-carboxylic acid in good yield (Scheme XI).



Scheme VII



Scheme VIII

ix. R. T. Pardasani *et al.*[46] have revealed a [3+2] cycloaddition reactions of cyclic secondary α-amino acids *viz*. L-proline and (R)-(−)-thiaproline with 5-methylthioisatin *via* azomethine ylide in the presence of dipolarophiles afford azabicyclooctane derivatives in moderate-to-good yields (Scheme XII).

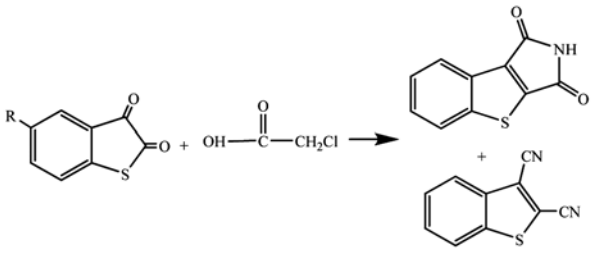
x. Condensation of benzo[b]thiophene-2,3-diones with *o*-phenylenediamines have been reported to give thionaphthaphenazines **(10)** whereas, non-*o*-phenylenediamines react with thioisatin to give **(11)**[47].

xi. Recently J. George, *et al*.[48] have accomplished a simple and efficient method for the synthesis of novel spirothiazolidinones by nucleophilic cyclocondensation of intermediate imine with mercaptoacetic acid is described. Computational studies have been performed to substantiate the proposed mechanism as well as to ascertain transition state of the system.

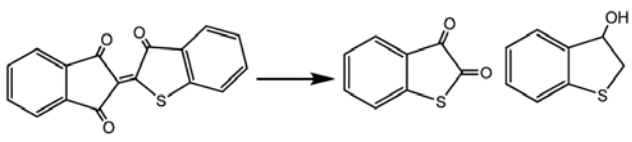
The entire reaction was carried out by the use of "Dean Stark Apparatus" and theoretical amount of water was removed azeotropically. The crude product was purified by column chromatography on silica gel with petroleum ether: chloroform (1:1) in 54-68% yield.

Synthesis and reactions of disulfides derivatives

Curt Wentrup, Harald Bender and Gerhard Gross[49] reported the synthesis of benzothiet-2-one in nearly quantitative yield (94%) as a pale-yellow solid by flash vacuum pyrolysis (FVP) of benzothiophene -2, 3-dione at 650°C. The product is isolated on a cold finger cooled with liquid nitrogen. Benzothietone reacts below −40 with methanol to give methyl



Scheme IX



Scheme X

o-mercaptobenzoate **(12)** in 85% isolated yield. The thiol **(13)** is completely oxidized to the disulfide **(14)** by prolonged stirring in the presence of air (Scheme XIII, Scheme IV).

Cleavage of C-S bond

When thioisatin derivatives were treated with alkali, cleavage of C-S bond occurs to form 2-mercaptophenyl glyoxalic acid which on treatment with chloroacetic acid in the presence of a base gives thiophenic dicarboxylic acid (Scheme XV)[50].

Oxidation will take place when benzo[b]thiophene-2, 3-diones treated with hydrogen peroxide and ammonia to afford 1, 2-benzoisothiazoles[51]. But, when oxidation is carried out with hydrazoic acid in sulphuric acid a dimer is formed from thioisatin.

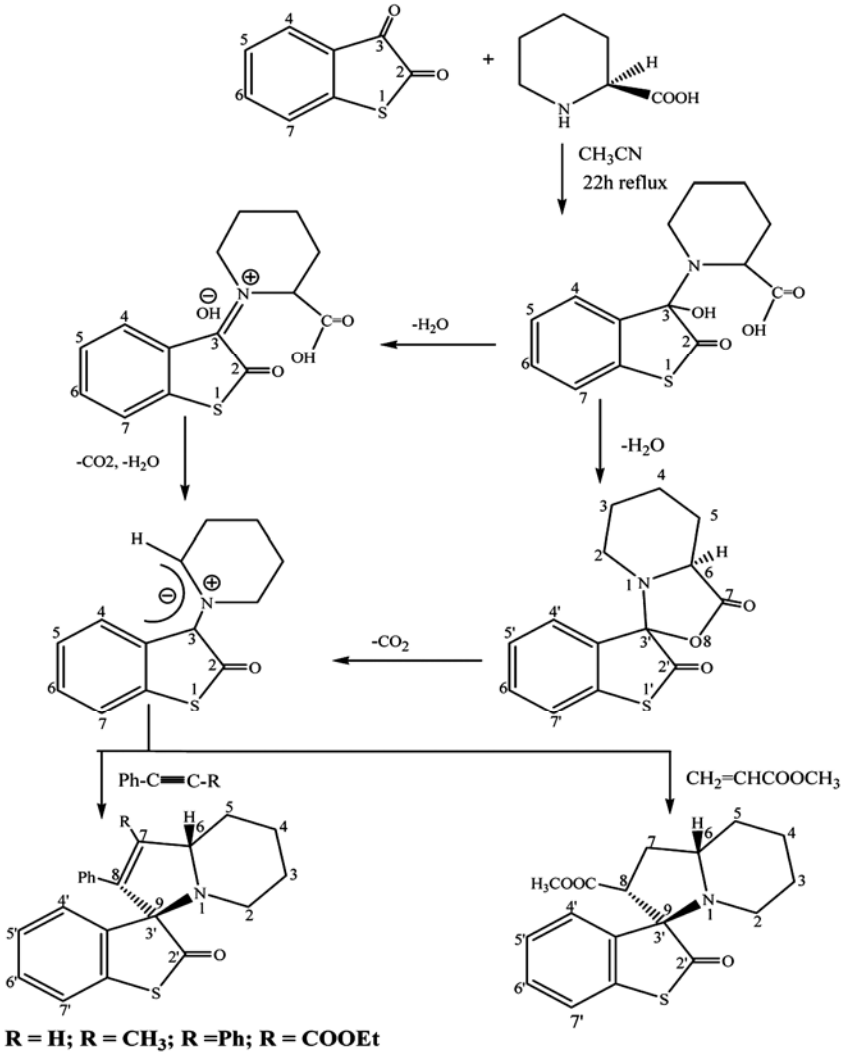
Benzo[b]thiophene undergoes ring opening and subsequent cyclization with ethyl cyanoacetate to give thiocoumarin derivatives which takes place in the presence of triethylamine[52].

Disulfide dimmer compounds[53] were synthesized by the treatment of thioisatin with diethylamine at 80°C for 3hr in nonpolar dry toluene (Scheme XVI) and kept at RT in open environment for one night. After trapping the solvent, rectangular shaped crystals were formed by air oxidation in high yield (72-75%) and purity (Figure 2).

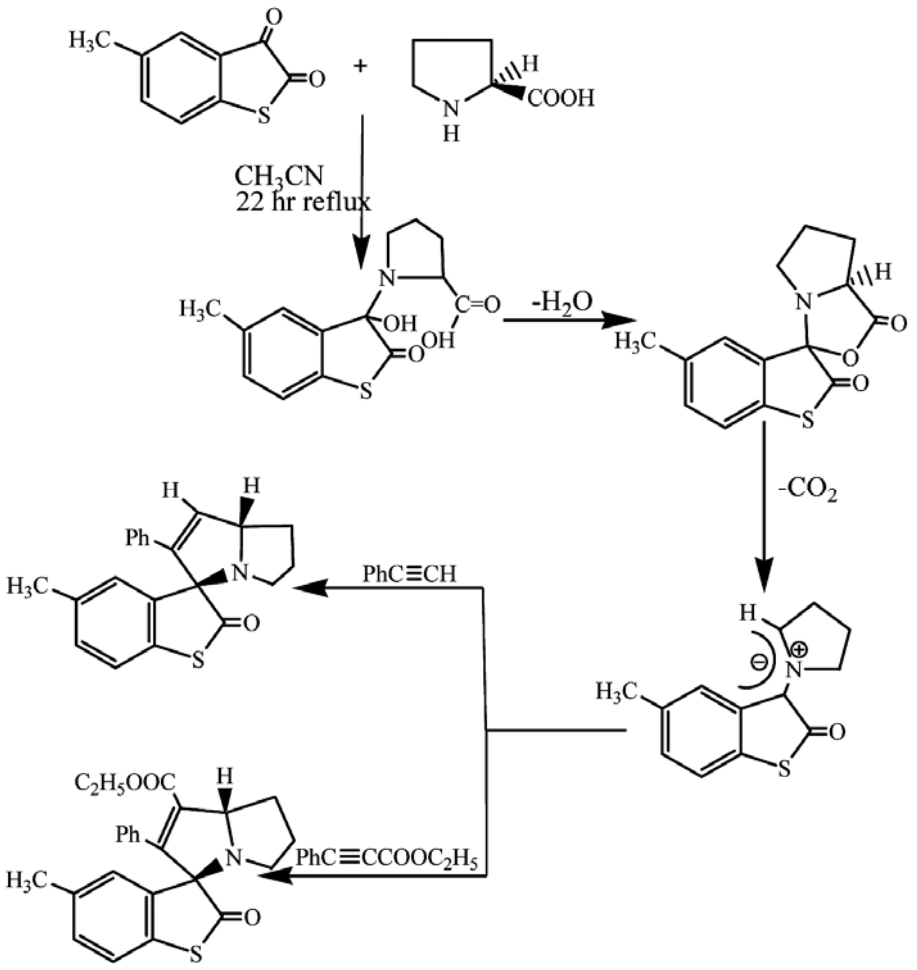
Disulfide derivatives reactivity of β- carbonyl functionality

Photocycloaddition reactions

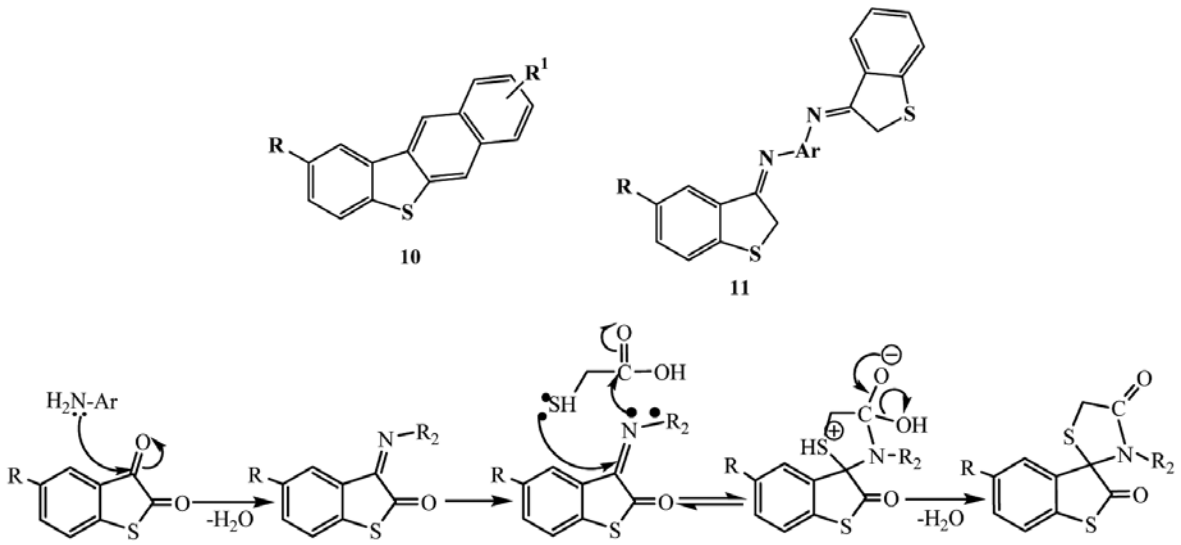
Cyclobutane[54], oxetanes[55] and thietanes[56,57] are synthesis by photo-cycloaddition of benzo[b] thiophene-2,3-diones to C=C, C=O and C=S bonds respectively to yield four membered cycles. However



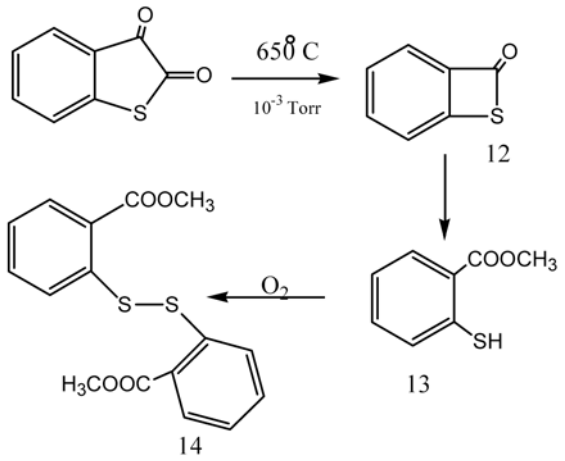
Scheme XI



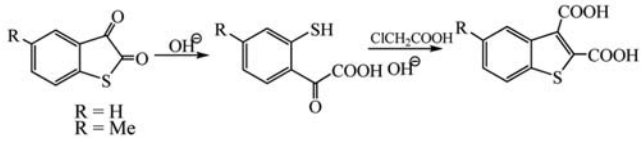
Scheme XII



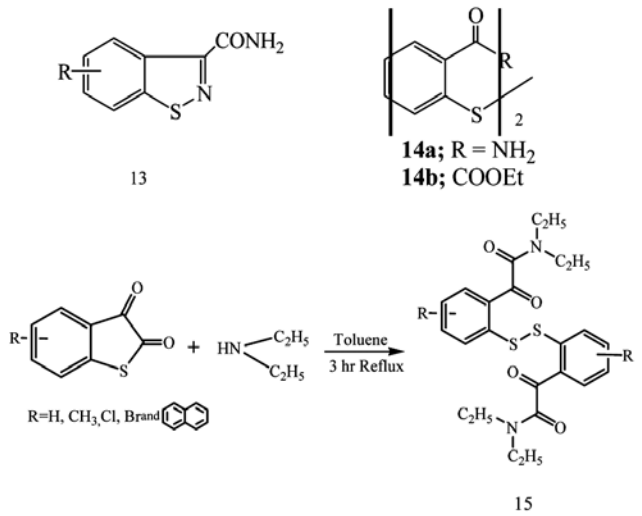
Scheme XIII — Mechanistic pathway for the formation of spirothiazolidinones



Scheme XIV



Scheme XV



Scheme XVI

similar [4+2] photocyclodddtions reactions with other olefins are less common. Examples are persented below.

Dioxene derivatives have been synthesized by photochemical reactions of benzo[b]thiophene-2,3-diones with 2, 3-dimethylbut-2-ene gave in excellent yields (Scheme XVII)[58].

Benzo[b]thiophene-2,3-dione with electron-rich and electron deficient alkenes have been described as [4+2] Photocycloadditions. Olefines preferentially add at both carbonyl groups to give the head-to-head [4+2] cycloadducts, *i.e.*, dioxanes only (Scheme XVIII)[59].

Syntheses of 3-(phenyl)-benzo[b]thiophene [2, 3-d][1,2] benzodiazocine derivatives[60] have been accomplished by the reaction of 3-phenacylidine-2-benzo[b]thiophene-2-ones with *o*-phenylene diamine. The photolytic reaction with *trans*-stilbene resulted in the exclusive formation of spiro{2_,5_,6_-triphenyl-2H-pyran-4,3}-benzo[b]thiophene-2-one derivatives

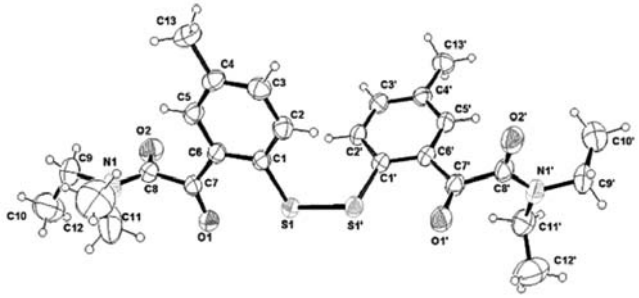
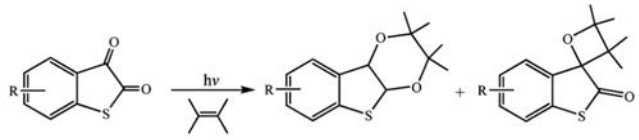
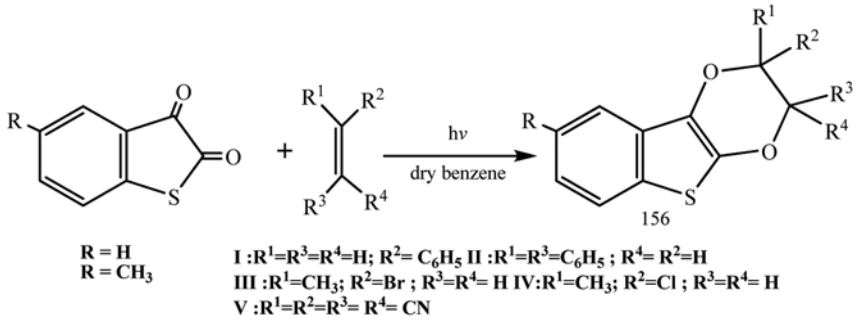


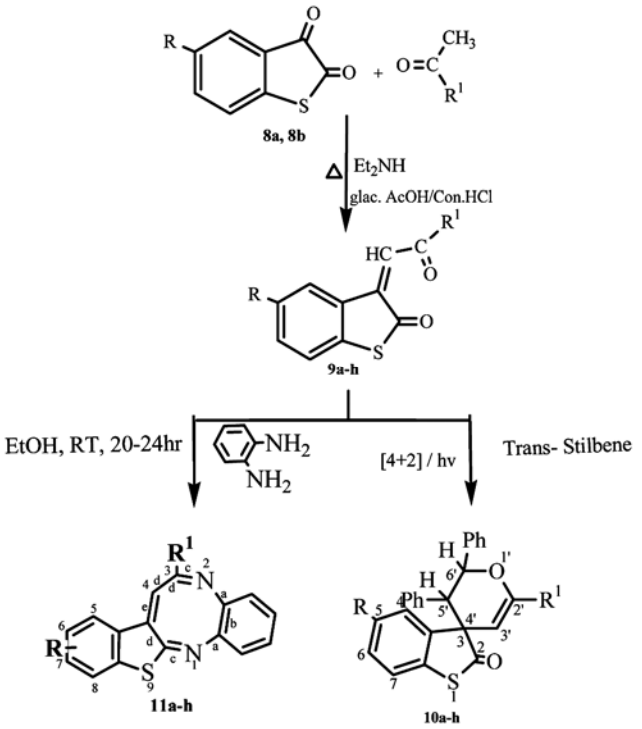
Figure 2 — An ORTEP view of the molecule of compound **15** with atom numbering. Displacement ellipsoids are drawn at 30% probability level



Scheme XVII



Scheme XVIII



Scheme XIX

(Scheme XIX). Theoretical calculations have been performed to study the mechanism and stereoselectivity of products. Good yield and broad scope of usable substrates of industrial relevance are other prominent features of the present methodologies.

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