



ISSN No: 0975-7384  
CODEN(USA): JCPRC5

*J. Chem. Pharm. Res.*, 2010, 2(6):167-170

## **A facile conversion of triazolobenzothiazole-3-thione to triazolobenzothiazole-3-ones by sulphur extrusion**

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### **ABSTRACT**

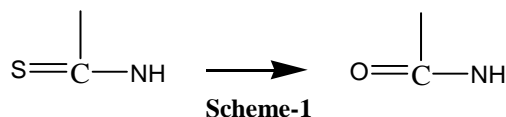
*Transformation of triazolobenzothiazole-3-thione to triazolobenzothiazole-3-one using active manganese dioxide where reagent displaced sulphur offering its own oxygen a unique feature. The isolated products have been characterized by spectral data and also authenticated by chemical method.*

**Keywords:** Triazolobenzothiazole-3-thione, triazolobenzothiazole-3-one, hydrazones.

### **INTRODUCTION**

In an endeavor to study the reaction for stability of 3-substituted triazolobenzothiazoles especially towards different oxidizing agent; it was noticed a facile conversion of thioamides to amides is taking place in good yields using active manganese dioxide. When dil. Nitric acid [1]

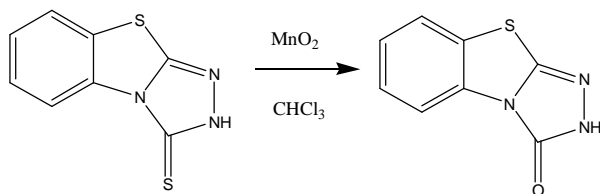
was used, 3-mercapto compound  $\text{N}=\text{C}(\text{R})\text{SH}$  was converted into  $\text{N}=\text{C}(\text{R})\text{H}$  parent compound. Here simple dethion [2] is taking place. For converting -thione to -one diverse synthetic methods are also available (scheme-1).



Using t-butoxide [3], sodium ethoxide, sodium hydroxide with halogen under phase transfer catalyst [4] trimethyl oxonium salt [5]. All being used with some degree of success. Recently

Maruda [6] and other used trichloro acetic anhydride. However the different reagents used for these conversions having varying degree of success as well as limitations due to side reactions. In the present case it is not simple elimination of sulfur but simultaneous introduction of oxygen in the molecule.

We now report reasonably simple and efficient method which enables to desulfurise amides to their oxo analogue in good yields using active manganese dioxide s-triazolothiazol-thione (scheme-2).



Scheme-2

This reaction is quantitative and occurs to the total excursion of thiazole ring oxidation. Surprisingly unknown new behavior of manganese dioxide appears to be of much interest. Variety of s-triazolo(3,4-*b*)benzothiazol-3-thione were smoothly converted into corresponding s-triazolo(3,4-*b*)benzothiazol-3-one.

## EXPERIMENTAL SECTION

### 1) General procedure for the synthesis of S-triazolo (3,4-*b*) benzothiazole 3-thione:

2-Hydrazino benzothiazole (0.82 gm) was taken along with potassium hydroxide (0.56 gm) in water (5 ml). To this carbon disulphide (1.9 gm) in ethanol (30 ml) was added. The mixture was heated to reflux on water bath for two hours. The residue was taken out after concentration in potassium hydroxide solution (20ml, 5%). The solution was stirred well and filtered. This solution on acidification yielded s-triazolo(3,4-*b*)benzothiazole 3-thione.

### 2) General procedure for the conversion of thioamide to amide using active manganese dioxide:

Thione (1gm) was dissolved in dry chloroform (25ml) and active manganese dioxide (4gm) was added to the reaction mixture. It was stirred at room temperature and monitored by TLC. After 14 hours the reaction mixture was filtered and the residue was washed with chloroform. The filtrate was concentrated. The product on crystallization yielded desired product

### 3) Authentication of the amide product obtained from manganese dioxide reaction with thione:

#### Procedure for the preparation of s-triazolo(3,4-*b*)benzothiazole-3-one by the condensation of 2-hydrazinobenzothiazole with urea.

2-Hydrazino benzothiazole (0.5 gm) was mixed with pre dried urea (0.4gm) in a round bottom flask and heated at 180-190<sup>0</sup>C for six hours on an oil bath. The contents were cooled and sodium hydroxide (30ml, 5%) solution was added. Filtrate on acidification with dilute hydrochloric acid gave s-triazolo(3,4-*b*)benzothiazole-3-one.

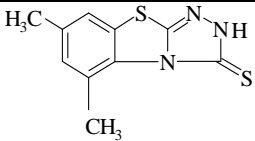
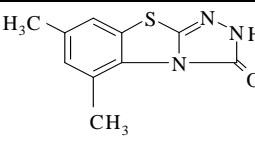
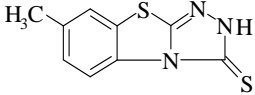
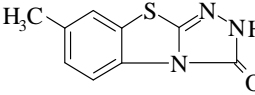
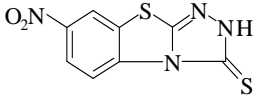
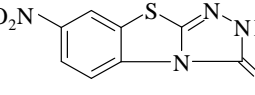
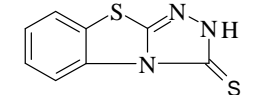
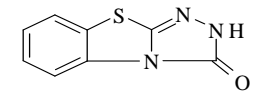
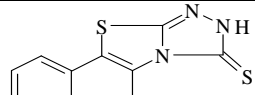
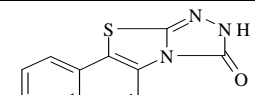
## RESULT AND DISCUSSION

All these reactions have been done at room temperature by shaking the mixture with oxidant (active manganese dioxide) in chloroform 12-16 hrs while monitoring the reaction by TLC. The corresponding amide has been obtained as the end product which was identified by spectral data and also its authentication by chemical method. Appropriate hydrazone compound was condensed with pre dried urea and the product is isolated [8].

When the reaction is carried out under inert atmosphere similar product was obtained. This proves that oxygenation is occurring through oxygen transfer from active manganese dioxide to thione substrate. The reaction reported is unique in the way reagent displaced sulfur offering its own oxygen.

The high yield and purity of product obtained compared with simple experimental procedure undoubtedly makes this method superior to some of the earlier known method m.p; yields and other detail refer table 1.

**Table-1: Conversion of thioamides to amides**

Sr. No.	Thioamide (A)	Amide (B)	Yield %	(A) m.p. <sup>0</sup> C	(B) m.p. <sup>0</sup> C	Hydrazino and urea condensation ( C ) m.p. <sup>0</sup> C
1			68	263	267	268
2			62	312	248	248
3			65	305	265	265
4			68	250	236	238
5			71	326	335	334

### Acknowledgement

Authors express their deep sense of gratitude to Dr. M. F. Rahman for his kind advice. Thanks are due to Dr. G. M. Kalamse, Principal, Science College, Nanded for encouragement.

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