

See discussions, stats, and author profiles for this publication at: <https://www.researchgate.net/publication/250470152>

# Mild and Efficient Method for Oxathioacetalization of Carbonyl Compounds

ARTICLE in CHEMINFORM · MAY 2007

Impact Factor: 0.74 · DOI: 10.1002/chin.200718044

---

CITATIONS

3

---

READS

2

3 AUTHORS, INCLUDING:



Adinath Majee

Visva Bharati University

92 PUBLICATIONS 1,000 CITATIONS

SEE PROFILE



Dr. Shrishnu Kumar Kundu

Jhargram Raj College

16 PUBLICATIONS 52 CITATIONS

SEE PROFILE

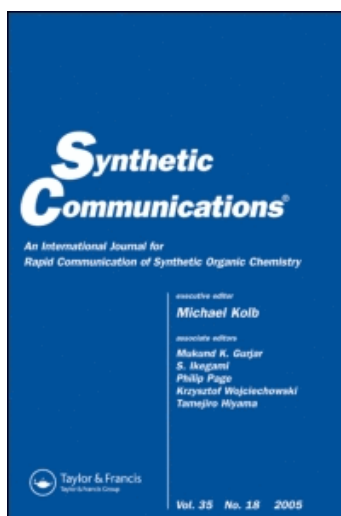
This article was downloaded by: [INFLIBNET India Order]

On: 22 June 2009

Access details: Access Details: [subscription number 909277354]

Publisher Taylor & Francis

Informa Ltd Registered in England and Wales Registered Number: 1072954 Registered office: Mortimer House, 37-41 Mortimer Street, London W1T 3JH, UK



## Synthetic Communications

Publication details, including instructions for authors and subscription information:

<http://www.informaworld.com/smpp/title-content=t713597304>

## Mild and Efficient Method for Oxathioacetalization of Carbonyl Compounds

Adinath Majee <sup>a</sup>; Shrishnu Kumar Kundu <sup>a</sup>; Samimul Islam <sup>a</sup>

<sup>a</sup> Department of Chemistry, Visva-Bharati University, Santiniketan, West Bengal, India

Online Publication Date: 01 December 2006

**To cite this Article** Majee, Adinath, Kundu, Shrishnu Kumar and Islam, Samimul(2006)'Mild and Efficient Method for Oxathioacetalization of Carbonyl Compounds',Synthetic Communications,36:24,3767 — 3770

**To link to this Article:** DOI: 10.1080/00397910600947783

**URL:** <http://dx.doi.org/10.1080/00397910600947783>

## PLEASE SCROLL DOWN FOR ARTICLE

Full terms and conditions of use: <http://www.informaworld.com/terms-and-conditions-of-access.pdf>

This article may be used for research, teaching and private study purposes. Any substantial or systematic reproduction, re-distribution, re-selling, loan or sub-licensing, systematic supply or distribution in any form to anyone is expressly forbidden.

The publisher does not give any warranty express or implied or make any representation that the contents will be complete or accurate or up to date. The accuracy of any instructions, formulae and drug doses should be independently verified with primary sources. The publisher shall not be liable for any loss, actions, claims, proceedings, demand or costs or damages whatsoever or howsoever caused arising directly or indirectly in connection with or arising out of the use of this material.

## Mild and Efficient Method for Oxathioacetalization of Carbonyl Compounds

Adinath Majee, Shrishnu Kumar Kundu, and Samimul Islam

Department of Chemistry, Visva-Bharati University, Santiniketan,  
West Bengal, India

**Abstract:** An efficient procedure for the protection of carbonyl compounds into the corresponding 1,3-oxathioacetal has been achieved using PAS as catalyst.

**Keywords:** 2-Mercaptoethanol, PAS, protection

### INTRODUCTION

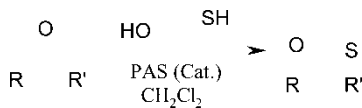
Oxathioacetals are prepared from the corresponding carbonyl compounds by reaction with 2-mercaptoethanol by an equimolar amount of Lewis acid such as  $\text{BF}_3 \cdot \text{OEt}_2$ <sup>[1]</sup> and  $\text{ZnCl}_2$ .<sup>[2]</sup> Other methods in the literature employ  $\text{TMSOTf}$ ,<sup>[3]</sup>  $\text{ZrCl}_4$ ,<sup>[4]</sup>  $\text{LiBF}_4$ ,<sup>[5]</sup>  $\text{HClO}_4$ ,<sup>[6]</sup> OTAB,<sup>[7]</sup> NBS,<sup>[8]</sup>  $\text{Sc}(\text{OTf})_3$ ,<sup>[9]</sup>  $\text{In}(\text{OTf})_3$ ,<sup>[10]</sup> TBAB,<sup>[11]</sup> and  $\text{Me}_2\text{S}^+\text{BrBr}^-$ <sup>[12]</sup> as catalysts. We have observed that carbonyl compounds and 2-mercaptoethanol react conveniently in the presence of polyaniline sulphate salt (PAS) as a catalyst to give the corresponding 1,3-oxathiolanes in good yields Scheme 1.


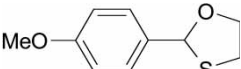
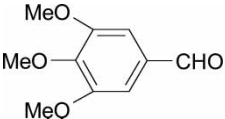
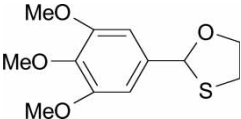
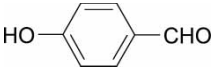
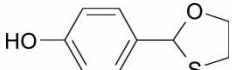
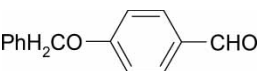

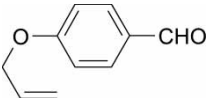
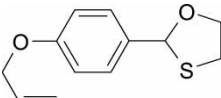
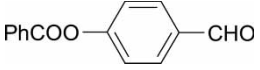
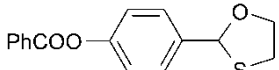
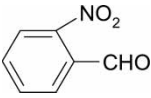
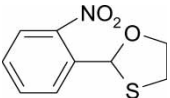

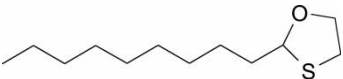
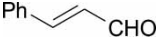
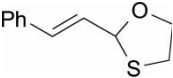
The results are shown in Table 1.

In a typical reaction procedure, the carbonyl compound (1 mmol), 2-mercaptoethanol (1.2 mmol), and a catalytic amount of PAS (0.01 mmol) in dichloromethane (3 mL) were stirred at room temperature as required for

Received in India May 4, 2006

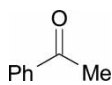
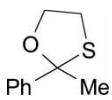
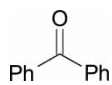
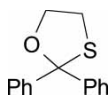
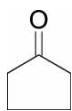
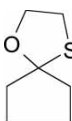
Address correspondence to Adinath Majee, Department of Chemistry, Visva-Bharati University, Santiniketan, West Bengal 731235, India. E-mail: adinathm@yahoo.com

**Scheme 1.****Table 1.** Preparation of oxathioacetals from the carbonyl compounds

Entry	Substrate	Time (h)	Product	Yield (%) <sup>a</sup>
1		3		72
2		5		60
3		4		60
4		4		70
5		4		65
6		2		75
7		4		70
8		6		65
9		4		75

(continued)

Table 1. Continued

Entry	Substrate	Time (h)	Product	Yield (%) <sup>a</sup>
10		6		60
11		4.5		65
12		4		60

<sup>a</sup>Yields are pure isolated product, characterized by IR and <sup>1</sup>H NMR.

completion as monitored by thin-layer chromatography, (TLC). Evaporation of the solvent from the reaction mixture under reduced pressure gives the crude product. The pure product is obtained by column chromatography.

## ACKNOWLEDGMENT

We are thankful to the Department of Chemistry, Visva-Bharati university and the DST-FIST program. We also express our sincere thanks to B. C. Ranu, HOD, Department of Chemistry, Indian Association for the Cultivation of Science, for his constant encouragement.

## REFERENCES

1. Wilson, G. E., Jr.; Huang, M. G.; Schloman, W.W., Jr. Facile synthesis of 1,3-oxathiolanes from ketones and i-mercaptoethanol. *J. Org. Chem.* **1968**, *33*, 2133.
2. Fallis, A. G.; Yadav, V. K. Cyclopentane synthesis and annulation: Intramolecular radical cyclization of acetals. *Tetrahedron Lett.* **1988**, *29*, 897.
3. Ravindranathan, T.; Chavan, S. P.; Dantale, S. W. Interconversion of oxathiolanes and carbonyls under essentially identical conditions. *Tetrahedron Lett.* **1995**, *36*, 2285.
4. Karimi, B.; Seradj, H. Zirconium tetrachloride (ZrCl<sub>4</sub>) as an efficient and chemo-selective catalyst for conversion of carbonyl compounds to 1,3-oxathiolanes. *Synlett* **2000**, 805.
5. Yadav, J. S.; Raddy, B. V. S.; Pandey, S. K. LiBF<sub>4</sub> catalyzed chemoselective conversion of aldehydes to 1,3-oxathiolanes and 1,3-dithianes. *Synlett* **2001**, 238.
6. Mondal, E.; Sahu, P. R.; Khan, A. T. A useful and catalytic method for protection of carbonyl compounds into the corresponding 1,3-oxathiolanes and deprotection to the parent carbonyl compounds. *Synlett* **2002**, 463.

7. Mondal, E.; Sahu, P. R.; Bose, G.; Khan, A. T. A useful and convenient protocol for interconversion of carbonyl compounds to the corresponding 1,3-oxathiolanes and vice versa employing organic ammonium tribromide. *Tetrahedron Lett.* **2002**, *43*, 2843.
8. Kamal, A.; Chouhan, G.; Ahmed, K. Oxathioacetalization, thioacetalization and transthoacetalization of carbonyl compounds by *N*-bromosuccinimide: Selectivity and scope. *Tetrahedron Lett.* **2002**, *43*, 6947.
9. Karimi, B.; Ma'mani, L. Scandium(III) triflate as an efficient and recyclable catalyst for chemoselective conversion of carbonyl compounds to 1,3-oxathiolanes. *Synthesis* **2003**, 2503.
10. Kazahaya, K.; Hamada, N.; Ito, S.; Sato, T. Indium trifluoromethanesulfonate as a mild and chemoselective catalyst for the conversion of carbonyl compounds into 1,3-oxathiolanes. *Synlett.* **2002**, 1535.
11. Ranu, B. C.; Das, A. Molten salt as a green reaction medium: Efficient and chemoselective dithioacetalization and oxathioacetalization of aldehydes mediated by molten tetrabutylammonium bromide. *Aust. J. Chem.* **2004**, *57*, 605.
12. Khan, A. T.; Sahu, P. R.; Majee, A. A highly efficient and catalytic synthetic protocol for oxathioacetalization of carbonyl compounds. *J. Mol. Cat. A* **2005**, *226*, 207.