

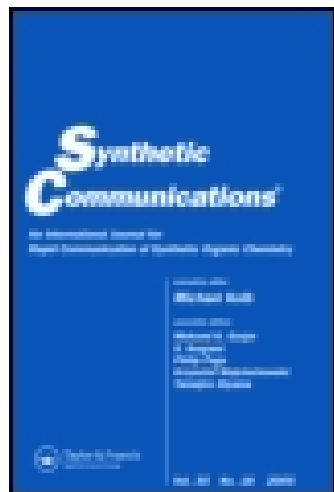
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## Mild and Efficient Method for Oxathioacetalization of Carbonyl Compounds

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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

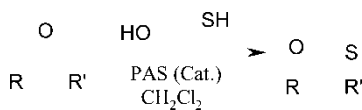
Oxathioacetals are prepared from the corresponding carbonyl compounds by reaction with 2-mercaptoethanol by an equimolar amount of Lewis acid such as  $\text{BF}_3 \cdot \text{OEt}_2$ <sup>[1]</sup> and  $\text{ZnCl}_2$ .<sup>[2]</sup> Other methods in the literature employ  $\text{TMSOTf}$ ,<sup>[3]</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.

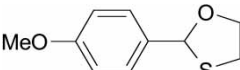
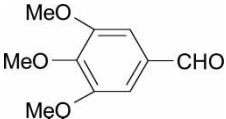
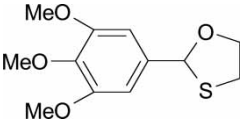
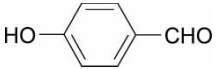
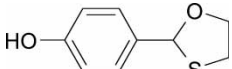
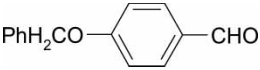
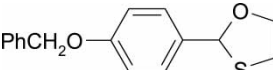
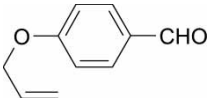
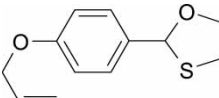
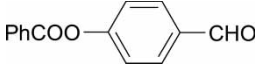
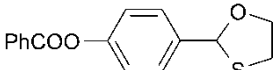
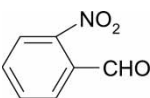
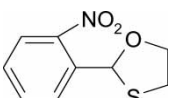
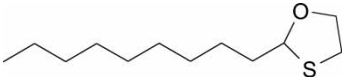
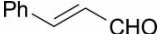
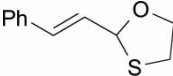
The results are shown in Table 1.

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

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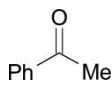
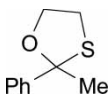
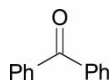
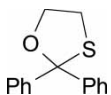
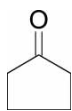
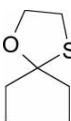
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**Scheme 1.****Table 1.** Preparation of oxathioacetals from the carbonyl compounds

| 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.

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