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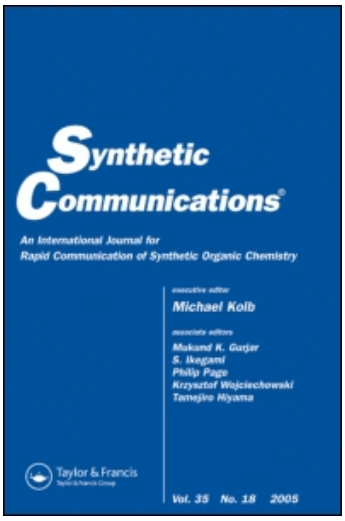

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Mild and Efficient Method for Oxathioacetalization of Carbonyl Compounds

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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-Marcaptoethanol, PAS, protection

INTRODUCTION

Oxathioacetals are prepared from the corresponding carbonyl compounds by reaction with 2-marcaptoethanol by an equimolar amount of Lewis acid such as $BF_3 \cdot OEt_2$[1] and $ZnCl_2$.[2] Other methods in the literature employ TMSOTf,[3] $ZrCl_4$,[4] $LiBF_4$,[5] $HClO_4$,[6] OTAB,[7] NBS,[8] $Sc(OTf)_3$,[9] $In(OTf)_3$,[10] TBAB,[11] and $Me_2S^+BrBr^-$[12] as catalysts. We have observed that carbonyl compounds and 2-marcaptoethanol 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-marcaptoethanol (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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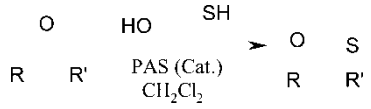

Scheme 1.

Table 1. Preparation of oxathioacetals from the carbonyl compounds

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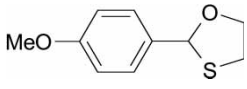
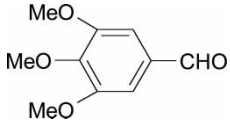
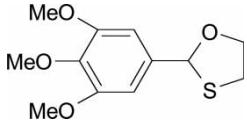
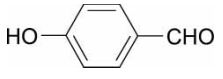
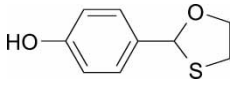
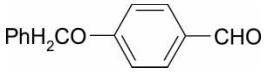
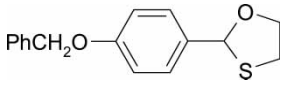
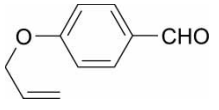
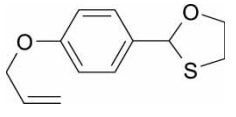
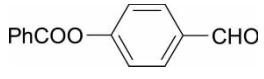
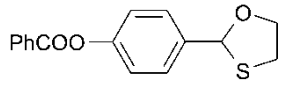
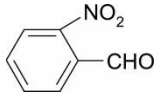
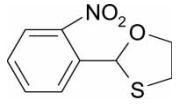
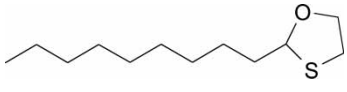
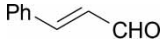
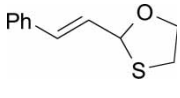
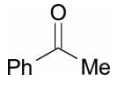
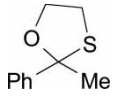
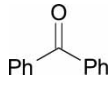
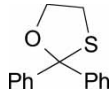
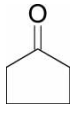
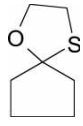
1		3		72
2		5		60
3		4		60
4		4		70
5		4		65
6		2		75
7		4		70
8		6		65
9		4		75

(*continued*)

Table 1. Continued

Entry	Substrate	Time (h)	Product	Yield (%)[a]
10		6		60
11		4.5		65
12		4		60

[a]Yields are pure isolated product, characterized by IR and ^{1}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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