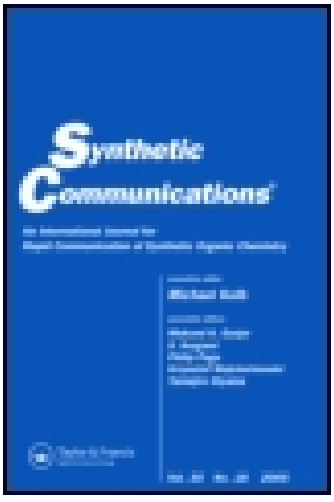

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One-Pot Synthesis of Acid Chloride from 1,2-Diols

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One-Pot Synthesis of Acid Chloride from 1,2-Diols

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Abstract: A simple and mild system for 1,2-diol cleavage has been developed using a combination of lead diacetate and *tert*-butyl hypochlorite at room temperature.

Keywords: acid chlorides, aldehydes, 1,2-diols, lead(II) acetate, *tert*-butyl hypochlorite

INTRODUCTION

Lead tetraacetate is widely used as an oxidizing agent in organic chemistry, however, it is moisture sensitive, hazardous to hande, and costly.[1] Thus our aim was to generate the lead tetraacetate in situ. In connection with our work on *tert*-butyl hypohalides,[2,3] we observed that lead(II) acetate reacts with *tert*-butyl hypochlorite to generate lead tetraacetate and can be used for cleavage of 1,2-diols to get the corresponding aldehydes. Further, it was noticed that with the use of excess *tert*-butyl hypochlorite, the corresponding acid chloride was formed.

Phenyl ethylene glycol was chosen for the reaction with lead diacetate and *tert*-butyl hypochlorite. A mixture of lead diacetate (2.1 mmol) and *tert*-butyl hypochlorite (2.0 mmol) in toluene was stirred at room temperature. Lead dichloride precipitated in 30 min, at which time phenyl ethylene glycol

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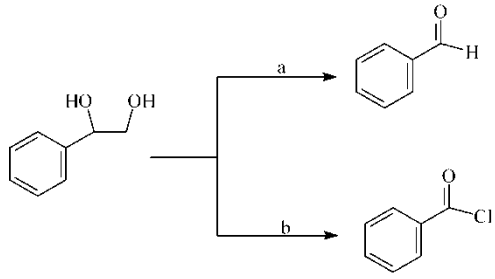
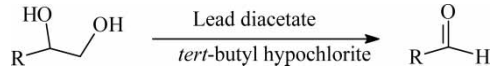


Scheme 1. Reagents and conditions: a) 1.0 mmol substrate, 2.0 mmol lead(II) acetate, 2.0 mmol *tert*-butyl hypochlorite; b) 1.0 mmol substrate, 2.0 mmol lead(II) acetate, 4 mmol *tert*-butyl hypochlorite.

Table 1. Oxidation of 1,2-diols using lead diacetate and *tert*-butyl hypochlorite[a]



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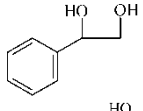
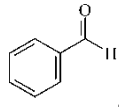
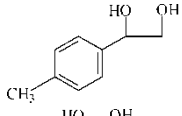
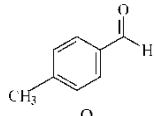
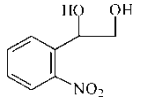
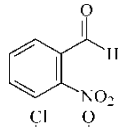
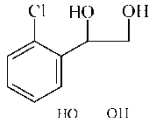
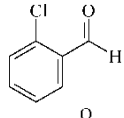
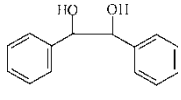
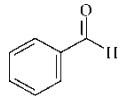
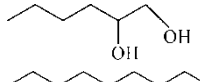
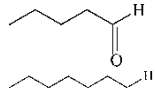
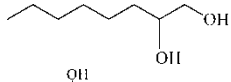
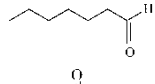
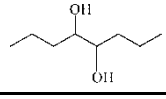
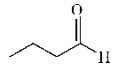
1			45	92
2			40	91
3			45	90
4			45	93
5			40	93
6			45	92
7			40	90
8			40	93

[a]All reactions were carried out at rt in toluene.
[b]Structures confirmed by IR, ^{1}H NMR, and mp/bp.
[c]Yields are of the isolated products.

(1.0 mmol) was added. After workup and purification by silica-gel chromatography using benzaldehyde, it was isolated in 94% yield (Scheme 1).

When the reaction was repeated with 4.0 mmol of *tert*-butyl hypochlorite, benzoyl chloride was formed. Encouraged by this observation, a variety of diols were subjected to the reaction with lead diacetate and *tert*-butyl hypochlorite in toluene, and results are given Tables 1 and 2.

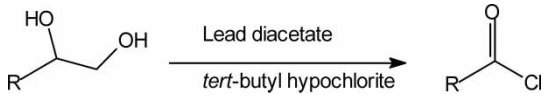
In conclusion, a simple, efficient, and mild one-pot method has been developed for the preparation of acid chloride from 1,2-diols.

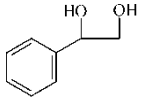
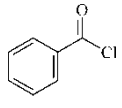
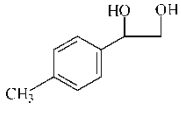
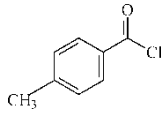
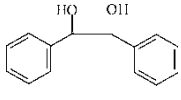
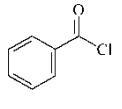
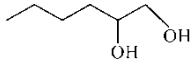
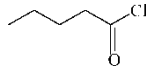
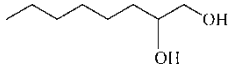
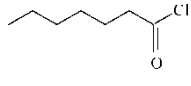
GENERAL PROCEDURE

Preparation of Aldehyde from 1,2-Diol

A mixture of lead acetate (8 g, 21 mmol) and *tert*-butyl hypochlorite (2.2 g, 20 mmol) in toluene (25 ml) was stirred at room temperature by adding a

Table 2. One pot acid chloride preparation from 1,2-diols using lead diacetate and excess *tert*-butyl hypochlorite[a]



Entry	Compound	Product[b]	Time (min)	Yield (%)[c]
1			45	92
2			40	91
3			45	93
4			40	92
5			40	90

[a]All reactions were carried out at rt in toluene.
[b]Structures confirmed by IR, ^{1}H NMR, bp.
[c]Yields are of the isolated products.

small quantity of benzoyl peroxide as an initiator. After 30 min, 1,2-octanediol (1.45 g, 10 mmol) was added and stirred for 15 min. The reaction mixture was filtered, the filtrate washed with saturated sodium bicarbonate and water (2 × 20 ml), dried on sodium sulfate, and evaporated to get the heptaldehyde.

One-Pot Preparation of Acid Chloride from 1,2-Diol

A mixture of lead acetate (7.6 g, 20 mmol) and *tert*-butyl hypochlorite (4.3 g, 40 mmol) in toluene (25 ml) was stirred at room temperature by adding a small quantity of benzoyl peroxide as an initiator. After 30 min, 1,2 octanediol (1.45 g, 10 mmol) was added and stirred for 15 min. The reaction mixture was filtered, and the filtrate was concentrated. The residue was distilled under vacuum to give heptanoyl chloride.

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