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ACETONIDE PROTECTION OF DIOLS USING IODINE AND DIMETHOXYPROPANE

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ABSTRACT
The protection and cleavage of isopropylidene acetals are important aspect in the chemistry of carbohydrates, nucleosides and alkaloids. As a result, numerous methods using acid catalysts have been developed. Herewith, we have reported novel method for protection of diol as an acetonide using iodine and dimethoxypropane. The ease of handling, cost and activity of the catalyst, good to excellent yields and neutral reaction conditions are some of the highlights of the reported method.

Keywords: Acetonide, Diol protection, Iodine, Dimethoxypropane (DMP), Green chemistry.

1. INTRODUCTION

A protecting group (PG) is introduced onto a particular functional group (FG) in a poly-functional molecule to block its reactivity under reaction conditions needed to make alterations elsewhere in the molecule. A decent protecting group should be readily, but selectively introduced to the desired functional group in a poly-functional molecule. It should be stable / resistant to the reagents used in consequent reaction steps in which the group being masked (protected) is desired to remain deactivated (protected) and it should be capable of being selectively removed under mild conditions.The commonly tackled functional groups in organic synthesis that are reactive to nucleophilic or electrophilic reagents whose selective transformation may present challenges do regularly require deactivation by masking with a protecting group. The most important method for the protection 1, 2 diols or 1, 3 diols is to convert them into a cyclic acetals or ketals.[1]The several methods are reported for the acetonide protection of diols by using acetone in presence of anhy. $FeCl_3$,[2]iodine,[3] $CuSO_4$,[4] and cation exchange resin.[5]Few methods are also described in the literature by suing 2, 2-dimethoxypropane in presence of $ZrCl_4$,[6] and *p*-TsOH.[7]Although a large number of methods using different catalysts are available for this purpose, the known methods are generally time consuming and under acidic conditions.Herewith, we are reporting a mild, efficient and eco-friendly method for protection of 1,2 or 1,3 diols as their acetonides by usingdimethoxypropane and iodine.

2. RESULTS AND DISCUSSIONS

In continuation with our efforts to develop the new methods for the synthesis of bioactive organic compounds,[8] we have demonstrated the new and efficient method for acetonide protection of diols. Accordance to our aim, we performed the reaction of 1, 2-diol (20 mmol) (**1a**) in presence of iodine (20 mol%) in dimethoxypropane (DMP)at room temperature to give the corresponding acetonide protected compound (**2a**) with 75% yield.To set the generality of reaction, we performed the reaction different diols in presence of catalytic amount of iodine in dimethoxypropane (DMP). All the reactions furnished the corresponding protected diols with 60-80% yields (**Scheme 1**). The structures of products obtained were confirmed by comparing melting points with reported in literature (**Table 1**).

Scheme-1

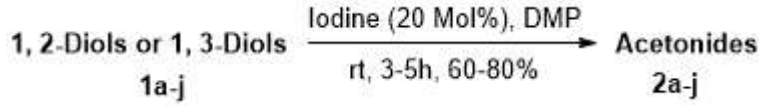

Table-1: Preparation of acetonide derivatives of 1,2 / 1,3-diols

1,2 or 1,3- Diols	Products	Time	Yield (%)
D-Glucose	2a	3	75
D-Mannitol	2b	4	60
D-Xylose	2c	5	65
D-Mannose	2d	4	65
Resorcinol	2e	4	80
Glycerol	2f	5	75
Propane 1,3-diol	2g	3	77
Catechol	2h	3	73
Cyclohexane 1,2-diol	2i	4	70
Cholestane 2,3-diol	2j	4	65

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3. EXPERIMENTAL

General procedure for the synthesis acetonides: In a solution of diol (20 mmol) in dimethoxypropane (DMP), iodine (20 mol%) was added. The reaction was stirred at room temperature. The progress of reaction was monitored by TLC. After completion of reaction, the product was extracted with ethyl acetate and purified by column chromatography to furnish the corresponding acetonides (2a-j) with 60-80% yields.

4. CONCLUSION

We have demonstrated a simple, efficient and green method for protection of diols. The neutral condition, good for acid sensitive starting materials and high yields are the key advantages of our protocol.

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