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MOLYBDENUM CATALYZED PROTECTIVE TETRAHYDROPYRANYLATION OF ALCOHOLS AND PHENOLS[+]

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Abstract : Molybdenyl(VI) acetylacetonate is an effective catalyst for the protective tetrahydropyranylation of alcohols and phenols.

Tetrahydropyranyl group is often the protective group of choice in peptide, nucleoside, carbohydrate and steroid chemistry because of the stability of the tetrahydropyranyl derivatives towards basic media, Grignard reagents and reactions involving oxidation and reduction by inorganic hydrides[1]. A wide variety of methods is available for this conversion[2]. Transition metal catalysts have not been used for this conversion so far. The low cost and ease of handling molybdenum catalysts prompted us to examine such a possibility *with molybdenyl* (VI) acetylacetonate complex.

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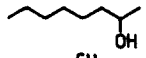
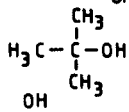
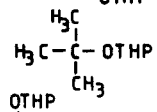
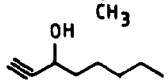
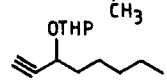
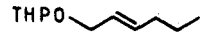
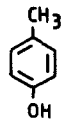
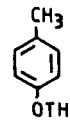
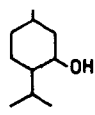
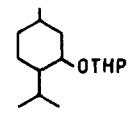
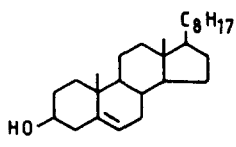
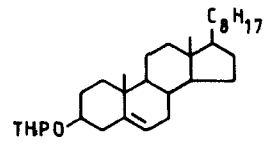
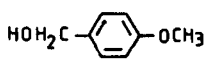
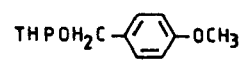
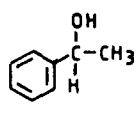
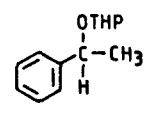
Table

Protective tetrahydropyranylation of alcohols catalysed by $MoO_2(acac)_2$

Entry	Substrate	Product	Yield (%)[a]
1			89
2			68
3			65
4			75
5			70
6			62
7			82
8			63
9			61
10			67
11			88
12			74
13			68

a - Isolated yield

We report here a simple and inexpensive tetrahydropyranylation of alcohols and phenols catalyzed by $MoO_2(acac)_2$ (2 mol%) in good yields (Table 1).

In summary, the mildness of the reaction conditions makes this catalyst very useful.

Experimental Section

Tetrahydropyranylation of Alcohols and Phenols; General Procedure:

In a typical experiment, a flask was charged with 0.260 g (2 mmol) of 1-octanol (Table 1, entry 1), 1 ml of 3,4-dihydro-2H-pyran and 0.013 g (0.04 mmol) of molybdenyl acetylacetonate in 10 ml of dry chloroform. The mixture was heated under reflux and argon with stirring whereupon the orange-yellow suspension turned blue. Progress of the reaction was monitored periodically by TLC. After 4h, the mixture was then cooled to room temperature and most of the chloroform was removed. The residue was then chromatographed on silica-gel to get the pure product (ethylacetate: hexane). Yield (0.380 g, 89%).

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