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Mg-FeCl₃-DMF-H₂O: An Inexpensive and Excellent System for Reduction of Aldehydes and Ketones

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Mg-FeCi₃-DMF-H₂O : AN INEXPENSIVE AND EXCELLENT SYSTEM FOR REDUCTION OF ALDEHYDES AND KETONES

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Aldehydes and ketones are reduced to correspondig alcohols in good yields at room temperature.

Literature survey reveals that numerours reagents ¹ are used for reduction of aldehydes and Ketones. Recently there are some more publications²⁻⁵ in this connection but those have some limitations and drawbacks. Zn-NiCl₂-DMF-H₂O system² and CoCl₂-Zn-THF system³ reduces only aldehydes and not ketones while Zn-CoCl₂-DMF-H₂O system⁴ reduces aldehydes in good yields but ketones in poor yields. FeCl₃-Zn-DMF-H₂O system⁵ reduces aromatic aldehydes and alicyclic ketones but not aromatic ketones. To overcome these drawbacks and limitations we explore a new, inexpensive and mild system which reduces aldehydes and ketones in excellent yields at room temperature (scheme).

Because of the ready access of the reagents used, mild reaction conditions, easy work-up, one step conversion and excellent yields we recommend this ecofriendly, simple, mild and inexpensive system for reductoin of aldehydes and ketones to corresponding alcohols. It does not reduces carbon-carbon double bond (entries 3 and 4) and nitro group (entries 5 and 6). The following alcohols were prepared in the laboratory (table). The progress of the reaction was monitered by TLC and products were characterised by comparison with authentic samples (physical constants, TLC, suitable derivatisation and spectral analysis (IR and PMR).

(Scheme)

TABLE

Entry	Aldehyde / Ketone	Times	Vield		
Entry		Time	Yield	B.P. / [M.P.]	
		h	%	obs	lit ^e
1.	Benzaldehyde	2.5	92	204-205	205
2.	Anisaldehyde	2.5	95	258	259
3.	Cinnamaldehyde	2.5	83	256-257	257
4.	Citral	2.5	85	230	229
5.	2-Nitrobenzaldehyde	2.5	72	[72]	[74]
6.	4-Nitrobenzaldehyde	1.5	91	[91]	[93]
7.	Furfuraldehyde	2.0	87	171	170
8.	4-Chlorobenzaldehyde	2.5	85	[74]	[75]
9.	Salicyaldehyde	2.75	71	[81]	[82]
10.	Cyclohexanone	3.0	89	161	160
11.	Cyclopentanone	3.25	87	139-140	139
12.	Acetophenone	3.5	85	201-202	203
13.	Benzophenone	3.5	81	[67]	[68]

GENERAL PROCEDURE FOR REDUCTION :

A mixture of $FeCl_{3}.6H_{2}O$ (25 mmole), Mg metal (0.6 g) and aldehyde / Ketone (5 m mole) in a mixed solvent DMF-H₂O (1:1,25ml) was stirred at r.t. After completion of the reaction (monitered by TLC) the reaction mixture was diluted with ether (30 ml)and filtered. The ether layer was washed with water (2 x 25 ml)and dried over anhydrous Na₂SO₄. Removal of solvent under reduced pressure gave alcohol which was further purified by crystallisation or distillation.

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