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Reduction of Carbonyl Compounds to Alcohols Using Ferric Chloride - Zinc-Dimethylformamide-Water System

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REDUCTION OF CARBONYL COMPOUNDS TO ALCOHOLS USING FERRIC CHLORIDE - ZINC-DIMETHYLFORMAMIDE-WATER SYSTEM

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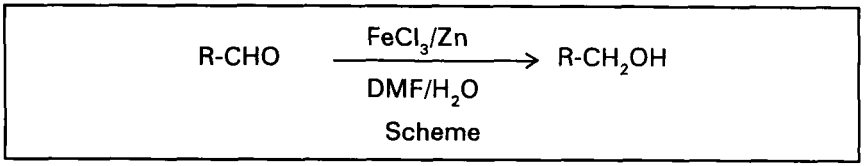
Carbonyl Compounds have been reduced into the corresponding alcohols in moderate to good yields at room temperature using Ferric Chloride-Zinc-Dimethyl-Formamide-Water System.

Numerous reagents[1-12] have been reported for reduction of aldehydes to the corresponding alcohols. We have now found that Ferric Chloride-Zinc-Dimethyl formamide-water is an efficient system for reduction of various aldehydes to the corresponding alcohols at room temperature in good yields (Scheme).

Because of ready availability of all the reagents used, mild reaction conditions, easy work-up and good yields we applied this simple method for reduction of aldehydes to the corresponding alcohols. Alicyclic ketones were also reduced but aromatic ketones were not reduced.The carbon carbon double bond was not reduced at all (e.g. Cinnamaldehyde).

The following compounds were prepared in the laboratory (See table). The progress of the reaction was monitered by thin layer chromatography and products were characterised by m.m.p., IR and PMR spectra.



Scheme

Table

Sr. No.	Aldehyde / Ketone	Time hr.	Yield (%)	B.P. Obs.	(°C) Lit.
1.	Benzaldehyde	1	81	203-204	205
2.	Anisaldehyde	1	90	258	259
3.	Cinnamaldehyde	1	80	249	250
4.	Salicyaldehyde	1	40	80*	82*
5.	o-Nitro Benzaldehyde	1.5	56	70*	71*
6.	Furfuraldehyde	1.5	85	171	170
7.	Cyclohexanone	1	60	160	160
8.	Cyclopentanone	1	42	138	139
9.	Acetophenone	10	NR		
10.	p-Bromobenzophenone	15	NR		
11.	Benzophenone	15	NR		

*M.P. = Melting point NR = No Reaction

EXPERIMENTAL

General Procedure for reduction :

A mixture of ferric chloride hexahydrate (15 m mole), Zinc dust (50 m mole) and benzaldehyde (5 m mole) was stirred at room temperature in a mixed solvent dimethyl formamide-water (1:1, 25 ml). After completion of the reaction (1 hr, monitered by TLC), the reaction mixture was diluted with ether (250 ml) and filtered. The filtrate was washed with water (2 x 100 ml) and dried over anhydrous sodium sulphate. The solvent was removed under reduced pressure to furnish the residue. It was purified by column chromatography using silica gel as an adsorbent and pet. ether-ethyl acetate (4:1) as an eluant. The yield of benzyl alcohol (81%, b.p. 203-204°) IR (KBr) : 3300, 1500, 1460 cm^{-1}; PMR ($CDCl_3$): δ 2:8 (s,1H), 4.62 (s,2H), 7.45 (s,Ar-5H).

In case of cyclohexanone and cyclopentanone extractions were carred out using methylene chloride instead of ether.

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