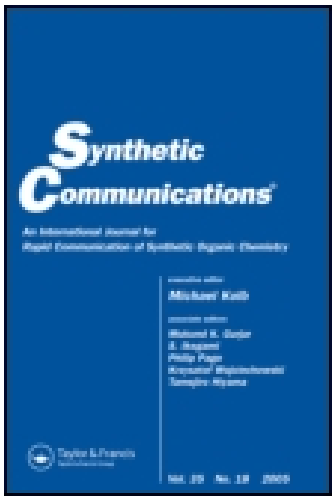

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SELECTIVE REDUCTION OF ALDEHYDES TO ALCOHOLS BY FeS-NH_4Cl-CH_3OH-H_2O SYSTEM*

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SELECTIVE REDUCTION OF ALDEHYDES TO ALCOHOLS BY $FeS\text{-}NH_4Cl\text{-}CH_3OH\text{-}H_2O$ SYSTEM*

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The System $FeS\text{-}NH_4Cl\text{-}CH_3OH\text{-}H_2O$ reduces selectively Aldehydes to corresponding alcohols in good yields.

Although a number of reagents are available in the literature[1] for the reduction of different functional groups, the development of mild, neutral and selective reducing system still attract a great deal of attention of organic chemists. In recent years, reagents based on iron find wide applications in organic synthesis because of their ready availability, ease of handling and low cost. Reduction of aldehydes to alcohols is an important reaction in organic synthesis and in structure determination of many natural products. The same is achieved by numerous reagents[2–13] in literature.

We report here that the $FeS\text{-}NH_4Cl\text{-}CH_3OH\text{-}H_2O$ is an efficient system for selective reduction of aldehydes to corresponding alcohols in good yields (scheme).

The chemicals required for present reducing system are easily available, cheaper and non-toxic. The system is mild neutral, ecofriendly and gives quite satisfactory yields which is useful addition to the existing systems to convert aldehydes to the primary alcohols (table). It does not reduce alicyclic ketones (Entries 12,13) as well as aromatic ketones (Entries 14,15).

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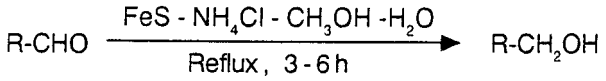

Scheme.

Table. Reduction of Aldehydes to Alcohols

Entry	Aldehyde/Ketone	Time (h)	Yield (%)	B.P./(M.P.) (°C)
1	Benzaldehyde	3.0	78	203–204
2	Anisaldehyde	4.0	70	257
3	4-Benzaldehyde	3.5	74	(61)
4	2-Chlorobenzaldehyde	6.0	65	(68)
5	4-Chlorobenzaldehyde	4.0	80	(74–75)
6	Furfuraldehyde	4.0	83	171
7	1-Napthaldehyde	3.5	74	(63)
8	Cinnamaldehyde	4.0	72	256–257
9	4-Bromobenzaldehyde	4.0	78	(77)
10	4-(*N*,*N*-Dimethylamino)-benzaldehyde	15	NR	–
11	Benzylideneacetophenone	15	NR	–
12	Cyclopentanone	15	NR	–
13	Cyclohexanone	15	NR	–
14	Acetophenone	15	NR	–
15	Benzophenone	15	NR	–

a. NR = No Reaction.
b. In case of salicylaldehyde and vanillin reaction mixture became highly coloured.

The carbon–carbon double bond also was not affected (Entries 8, 11). The following compounds were reduced in the laboratory (table). The progress of the reaction was monitored by TLC and products were characterised by comparison with authentic samples,[14] suitable derivatisation and spectral analysis (IR, PMR).

EXPERIMENTAL (GENERAL PROCEDURE FOR REDUCTION)

Aldehyde/Ketone (10 mmol) was dissolved in a mixture of methanol (30 ml) and water (10 ml). To this commercial powdered iron sulphide

(50 mmol) and ammonium chloride (50 mmol) were added. It was refluxed in water bath (TLC monitoring, see table). When the reaction is over, residue was filtered, washed with methanol (2×5 ml). The solvent was removed from combined filtrate and washings by distillation under reduced pressure and residue was extracted with methylene chloride (3×30 ml). The combined organic layer was washed with water (3×30 ml) and dried over anhydrous sodium sulphate. Removal of solvent under reduced pressure furnished an alcohol which was further purified by distillation.

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