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## An efficient and environmentally friendly wolf-kishner reduction catalyzed by strong base anion-exchange resin

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### ABSTRACT

Wolf-Kishner reduction of aldehydes and ketones has been carried out by using strong anion-exchange resins (Amberlite IRA 400, Amberlyst A26 and Indion 820) as catalysts. The reactions are simple, rapid and gives high yields. © 2008 Trade Science Inc. - INDIA

### KEYWORDS

Anion-exchange resins;  
Aldehydes;  
Ketones;  
Reduction.

### 1. INTRODUCTION

Wolf-Kishner reduction is well-known reaction discovered by Wolf-Kishner, which involves reduction of carbonyl group to methylene group in a molecule using hydrazine in ethane diol as a solvent, catalyzed by base NaOH or KOH under reflux condition<sup>[1]</sup>. The reduction of an aldehyde or ketone to alcohol can be carried out by alkali metals as well as complex metal hydrides such as LiAlH<sub>4</sub> or NaBH<sub>4</sub><sup>[2]</sup>. The synthesis and initial applications of LiAlH<sub>4</sub> for reduction of organic compounds were reported by Schlesinger and co-workers<sup>[3]</sup>. Lithium aluminium hydride rapidly reduces most carbonyl compounds, including aldehydes and ketones<sup>[4]</sup>, carboxylic acids, acid anhydrides, acid chlorides, esters, lactones, amides, carbamates, imides and lactams<sup>[5]</sup>. The reduction can also be achieved by several alkali and transition metals<sup>[6]</sup>. A classical reduction that involves metal is the Meerwein-Ponndorf-Verley reduction<sup>[7]</sup>.

Basic anion-exchange resin is a type of solid base.

It has been widely used in synthesis of organic chemistry<sup>[8-12]</sup>. To our knowledge; there is no report of using strong base anion-exchange resin as a catalyst in the Wolf-Kishner reduction. Thus we wish to report Wolf-Kishner reduction catalyzed by strong base anion-exchange resin. The catalyst can be regenerated by activation i. e. by treatment with 10% NaOH.

### 2. EXPERIMENTAL

All the reagents were purified by methods reported in literature<sup>[13]</sup>. We have carried out Wolf-Kishner reduction of aldehydes and ketones by using strong anion-exchange resins such as Amberlite IRA 400, Amberlyst A26 and Indion 820 in presence of hydrazine hydrate and ethane diol solvent under reflux and stirring conditions. Strong anion-exchange resins were prepared as follows. The original resins were washed with deionised water, then exchanged with 10% NaOH. Finally washed with deionised water until it was neutral, dried at room temperature and stored in desiccator un-

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til use. All the products were characterized by their physical constants with literature data.

### General procedure for strong anion-exchange resin catalyzed wolf-kishner reduction

A mixture of aldehyde or ketone (10 mmole), hydrazine hydrate (10 mmole), strong anion-exchange resins (10 g) and 25 ml of ethane diol were refluxed with stirring until the reaction was completed. Different aldehydes and ketones required 50-65 min. for completion of reaction. The progress of reaction was monitored by TLC (Hexane: ethyl acetate, 9:1). After completion of reaction resin was filtered off. The resin

was washed with water (3X 10 ml). Then product from filtrate was extracted with ether.

After this ether was evaporated and further purification was achieved by distillation under reduced pressure. All the products obtained are listed in TABLE 1.

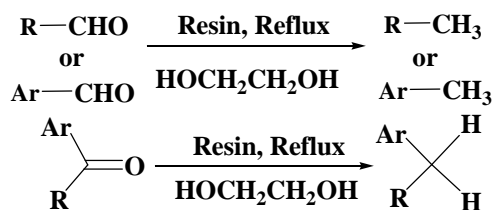
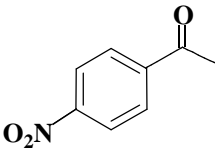
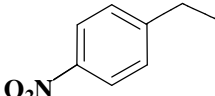
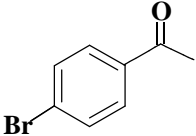
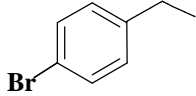


Figure 1

TABLE 1 : Wolf-Kishner reduction of aldehydes and ketones

	Substrate	Product	Rea. <sup>n</sup> time (min.)	Yield (%)			b. p. °C	
				Amberlite IRA 400	Amberlyst A 26	Indion 820	Obs.	Lit.
1			55	82	92	86	110	110
2			60	81	90	87	265	264
3			55	80	88	85	135	136
4			50	78	89	80	81	81
5			60	77	84	82	226	225
6			55	75	87	79	238	238
7			65	78	85	82	196	197
8			60	82	88	86	218	218
9			60	85	90	87	159	158

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	Substrate	Product	Rea. <sup>n</sup> time (min.)	Yield (%)			b. p. °C	
				Amberlite IRA 400	Amberlyst A 26	Indion 820	Obs.	Lit.
10			55	89	92	90	205	204
11			55	83	90	88	245	245

### 3. RESULTS AND DISCUSSION

In conclusion we have developed a practical Wolf-Kishner reduction procedure using strong base anion-exchange resin as a catalyst (**SCHEME 1**). By comparing the yields, using different anion-exchange resin as a catalyst for Wolf-Kishner reduction, it is found that Amberlyst A26 is better catalyst as compared to other resins. Thus the order of reactivity of the three basic anion-exchange resins is as follows.

Amberlyst A26 > Indion 820 > Amberlite IRA 400

### 4. ACKNOWLEDGMENTS

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