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Research Article

A simple Synthesis of Substituted 1,3-Dioxanes

GVR. Sharma* and KRK Prasad

Department of Chemistry, GITAM Instititute of Technology

GITAM University, Rushikonda, Visakhapatnam, Andhra Pradesh, India.

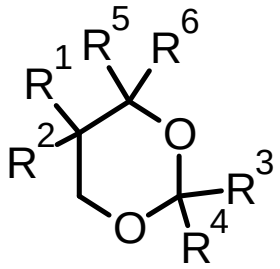
ABSTRACT

A simple synthetic method for the synthesis of Novel substituted 1,3-Dioxanes is reported in this communication. Products find extensive use as intermediates in Pharmaceutical, Cosmetics, and Perfumary industry.

Keywords: Dioxanes, diols, Synthesis.

INTRODUCTION

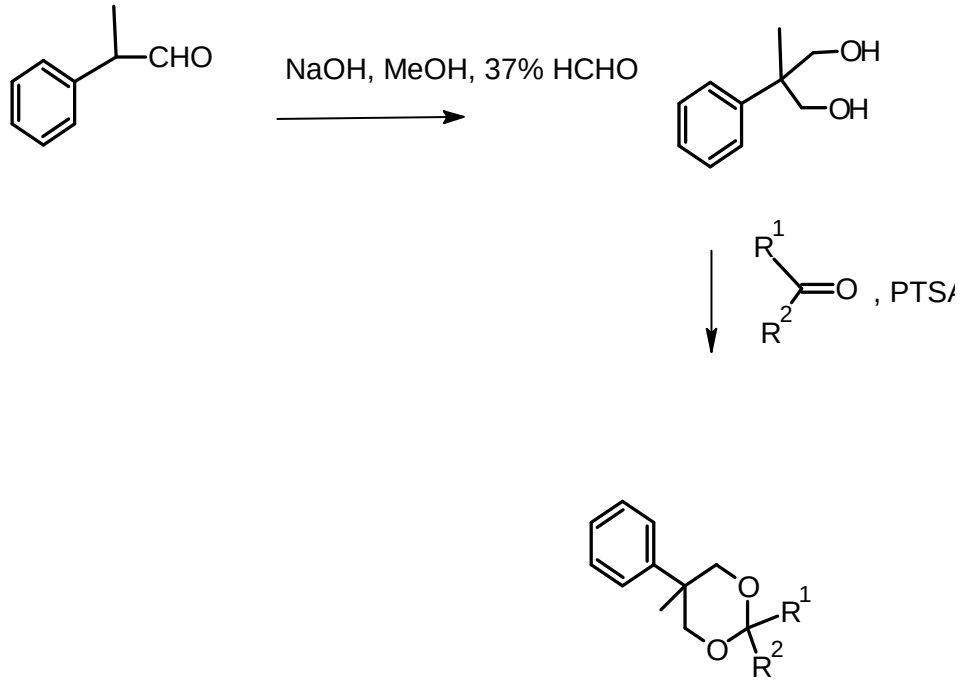
Substituted and unsubstituted 1,3-dioxanes find extensive application in Organic synthesis, as intermediates in pharmaceutical industry, cosmetic and flavor industries etc. [1-4] Several reports are available in the literature which describe methods to prepare differently substituted 1,3-dioxanes. However, those methods may not be generalised in many occasions. We have been focusing our studies on the synthesis and application of differently substituted 1,3-dioxanes and we found the method described in this communication is very simple and expected to find extensive use in organic synthesis, pharmaceutical and cosmetic industry. The general formula of a 1,3-dioxane is given below and a few reports involving the synthesis of these 1,3-dioxanes are mentioned in the references.[1-4]



R1, R2, R3, R4, R5, R6 represent atoms or groups such as H, alkyl, aryl, alkenyl, Nitro, Bromo etc.

RESULTS AND DISCUSSION

Our study started with alpha substituted aldehydes such as 2-phenyl propionaldehyde which when treated with aqueous formaldehyde in the presence of a base undergoes alpha formylation to give a dialdehyde which was in situ subjected to reductive conditions and provides the required diol in good yields. The diol is then reacted with ketones or aldehydes to form 1,3-dioxanes. The synthetic route is rather simple and provides the substituted 1,3-dioxanes in good to very good yields. All the products were characterized by spectral data such as 1HNMR, IR, and Mass. The reaction sequence involved is shown in the following scheme.



R^1 = H, Me; R^2 = Me, Et, n-Propyl, isobutyl

EXPERIMENTAL

Reagents and chemicals are obtained from commercial sources and used as such without purification. 1HNMR (Bruker 400 MHz) and Mass analysis was carriedout for all the intermediates and final compounds.

General Procedure from Aldehyde to Dioxane

A) Preparation of 2-methyl-2-phenyl-1,3-propane diol

400 ml of methanol and 87 gr of NaOH are given into a 1 ltr 4 necked flask equipped with a mechanical stirred, thermometer, addition funnel , cooling bath . 200 gr of 37% formaldehyde is added within 1hr at room temperature. And then 100gr of 2-methyl-2-phenyl propanal is added to reaction mixture within 2 hrs at 25-30°C ., after addition of Aldehyde stirred for 2hrs at RT. Adjust the PH-6, and distilled the solvent under atmospheric pressure. Cool the mixture and neutralized with $NaHCO_3$ solution. Filtered with sodium bisulphate and then distilled it to give the 86 gr of diol.

B) Preparation of Dioxane

85 gr of above diol , 350 ml of acetone ,1 gr PTSA are taken into a 500 ml flask equipped with a mechanical stirrer, thermometer, and addition funnel. The mixture is stirred for 2 hrs at room temperature .and then 10 gr of solid $NaCO_3$ is added, distilled the acetone, and given the water wash to neutral. Then filtered with Na_2SO_4, distillation to get 89 gr of 2,2,5-trimethyl-5-phenyl-1,3-Dioxane.

Similarly 2-Ethyl-5-methyl-5-phenyl-1,3-dioxane, 2-isopropyl-5-methyl-5-phenyl-1,3-dioxane, 2-Propyl-5-methyl-5-phenyl-1,3-dioxane are prepared and characterized by 1HNMR, Mass etc.

1HNMR in $CDCl_3$: δ 0.9 (s, 3H, Me), 1.0 (s, 3H, Me), 1.1 (s, 3H, Me), 3.6-4.4
(4H, CH2-O), 7.0-7.6 (m, 5H, Ar)
Mass: 206 (M^+).

CONCLUSION

In conclusion, we have reported in this communication a simple and brief synthetic methodology for the synthesis of substituted 1,3-dioxane which find extensive application in pharmaceutical, Cosmetic and flavor industry apart from its potential use in organic synthesis. Further work is in progress which will be communicated in an appropriate journal.

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