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SYNTHESIS OF 2,5-DIMETHYL-5-ACETYL-1,3-DIOXANE

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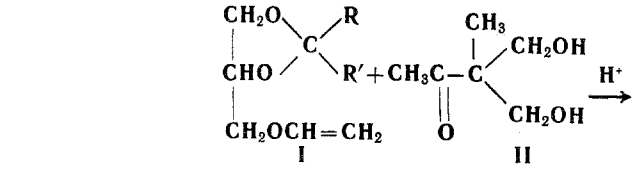
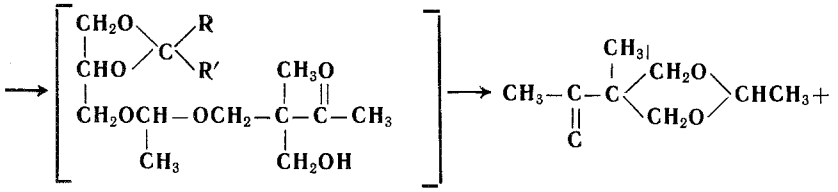
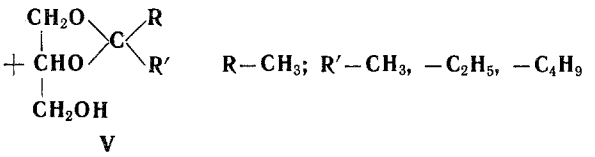
Simple vinyl ethers add ketoalcohols to give acetals containing a carbonyl group [1]. Continuing research on the chemical changes undergone by 4-vinylhydroxymethyl-2,2-dialkyl-1,3-dioxolanes, we have investigated their behavior with respect to ketoglycols, and this led to preparation of cyclic acetals containing the keto group:



I II



III IV



V

The exothermic reaction between I and II was accompanied by considerable tar formation from the reaction products. So it was run at −5° to −10° C, in the presence of traces of mineral acids. The yields of cyclic acetals containing the carbonyl group amounted to 75-85%.

4-Vinylhydroxymethyl-2,2-dimethyl-1,3-dioxolane and 2-methyl-2-acetyltrimethyleneglycol gave a 78.3% yield of 2,5-dimethyl-5-acetyl-1,3-dioxane (IV), bp 67° C (4 mm), n_D^{20} 1.4443, d_4^{20} 1.0493. Found: C 60.59, 60.39; H 9.25, 9.31%; MR 39.97. Calculated: C 60.73; H 8.92%; MR 40.169. The IR spectrum showed absorption at 1712 cm^{-1} (> C=O); 1017, 1039, 1053, 1106 cm^{-1} (acetal group).

The yield of 4-methylol-2,2-dialkyl-1,3-dioxolane (V) was 80-90%.

REFERENCES

1. M. F. Shostakovskii, V. M. Vlasov, P. I. Grenovskii, and V. G. Bologova, ZhOKh, 33, 4021, 1963.

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