

Acetylation by Keten of Phenols supported on Solid Adsorbents

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Summary Even hindered phenols, when adsorbed on solids such as silica gel and alumina, add to keten without heating or sulphuric acid catalyst.

ACETYLATION of phenols with keten requires both acid catalyst and high temperatures.¹ We found, however, that phenols on a solid adsorbent readily react with keten. To a solution of 1 g of phenol was added 15 g of silica gel powder, followed by evaporation of the solvent. The silica gel was packed in a glass column, then keten was introduced in a stream of inert gas at room temperature. After com-

pletion of the reaction, the product was eluted with a solvent. Purification by column chromatography gave 1.24 g of phenyl acetate. Alumina, titanium oxide, zinc oxide, magnesium oxide, celite, and charcoal were also found to be effective adsorbents for the acetylation at 0 °C and -78 °C. 2-Isopropylphenol, 2-t-butylphenol, 2,6-dimethylphenol, 2,6-di-isopropylphenol, 2,6-di-t-butylphenol, 1-naphthol, 2-naphthol, and hydroquinone adsorbed on alumina gave the corresponding acetates quantitatively at 0 °C.

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¹ R. N. Lacey, 'Advances in Organic Chemistry, Methods and Results,' eds. R. A. Raphael, E. C. Taylor, and H. Wynberg, Interscience, New York, 1960, vol. 2, p. 213.