

The Mechanism of Intramolecular Phosphine-Promoted Knoevenagel Redox Reaction

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DESCRIPTION

The carbon-carbon bond development is the foundation in the formation of numerous compounds utilized in products, like drugs, insect poisons and pesticides, and as intermediates for natural product generation. In 1894, the main response of formaldehyde with diethyl malonate, known as the Knoevenagel buildup, was discovered. These days, the Knoevenagel buildup is viewed as a nucleophilic expansion of a C-H acidic compound to a carbonyl group of an aldehyde or ketone to form a double bond under elimination of one equivalent of water. In recent years, a wide extent of various substrates, as well as various bases and reaction conditions, has been described. Extensive application is the utilization of piperidine as a base. In some cases, the reaction is completed at room temperature, and in further cases, the utilization of acidic acid as an added substance close to a nonpolar solvent like benzene or toluene under reflux conditions was laid out.

However, the majority of these transformations are two-step reactions, where the decrease, with an external reducing agent, happens after the change. Strategies involving inorganic reducing agents in combination with the Knoevenagel, similar to zinc in acidic acid and sodium borohydride, as well as enzymatic reduction and hydrogenations with palladium. An external triphenylphosphine particle was applied to perform a phospho-Michael expansion prompting a zwitterionic enolate, followed by P-O bond development and C-P bond cleavage to give reduced aldol compounds. Thus, we report that this sort of component can also be found in an intramolecular phosphine-promoted Knoevenagel redox response, where the phosphine oxide moiety remains in the molecule.

During analysis in the field of a Knoevenagel reaction with 4,4,4-

trifluoro-1-(thiophen-2-yl)butane-1,3-dione (TTA) and 2-(diphenylphosphanyl)benzaldehyde within the presence of piperidine, acidic acid gives new phosphine ligands. It is found that the decrease of the keto capability in the α -position to the CF₃ group of TTA, though the phosphine atom of the triphenylphosphine moiety was changed to the phosphine oxide with a yield of 39%.

The reaction was completed under Dean-Stark condition with dry toluene. The product was formed in equivalent yield, showing that the water *in situ* created by the Knoevenagel condensation takes part quickly in the reaction. To examine this strange kind of reaction, various ketones, acids, bases and reaction conditions were investigated in the transformation and the subsequent products were analyzed with ¹H-, ¹³C and ³¹P-NMR spectroscopy. Furthermore, the reaction was done under dry and deoxygenated conditions and was followed by *in situ* ³¹P-NMR. The spectra demonstrate the formation of the phosphine oxide without oxygen from the air, which prompts the suspicion that water formed in the condensation reaction is the oxygen source for the oxidation of the phosphine.

It revealed an intramolecular Knoevenagel redox reaction, which is promoted by intramolecular oxidation of a phosphine source, acquired after a regular Knoevenagel product formation. The mechanism of this reaction we performed different reactions with a variation of diketones, aldehydes, acids and bases and analyzed the products by means of ¹H-, ¹³C- and ³¹P-NMR spectroscopy. The outcome shows that the electronic structure of the diketone impacts the construction of the product. The utilization of an electron-withdrawing group prompts a decrease in the keto group, while electron-donating substituents of the diketone lead to the decrease of the double bond formed by the Knoevenagel reaction.

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