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Synthesis and Mechanism Studies on Amide Bond Formation by Hexamethylphosphoramide (HMPA)

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Synthesis and Mechanism Studies on Amide Bond Formation by Hexamethylphosphoramide (HMPA)

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In this paper, hexamethylphosphoramide (HMPA) was used as the organic phosphorus coupling reagent for the synthesis of amide bond. The reaction mechanism was tracked and investigated through ³¹P NMR.

Keywords Hexamethylphosphoramide (HMPA); mechanism; triphosgene

Protein and peptide with the amide bond backbone play important role in the organism. For the production of the amide bond, there are several known methods, such as Yasutsugu Shimonishi method,¹ N-carboxyl anhydride (NCA) method,² and DCC-HOSu method.³

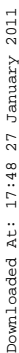
Appel had applied the organic phosphorous coupling reagents (e.g., triphenylphosphine) for the synthesis of peptide derivatives.^{4–7} In this article HMPA was used as amide bond coupling reagent, and the mechanism was studied.

The reaction was traced by ³¹P NMR (Figure 1).

Triphosgene was dissolved in solvent (anhydrous dichloromethane) and was dropwised to a stirred carboxylic acid and HMPA (a) in anhydrous dichloromethane at 0°C, the phosphinic carboxylic mixed anhydride (b) was formed. And then the above solution was dropwised to

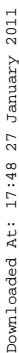
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