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Use of amino acids in enhancing the synthesis of N-phenylbenzylamine under microwave radiation

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Ullmann condensation is one of the most important organic reactions, which involves a Cu mediated aromatic nucleophilic substitution reaction. Classical Ullmann condensation had been modified by the addition of N,N- or N,O- bidentate ligands, where the ligands help the metal ions to increase the catalytic ability. Similarly, the rate of reaction can be enhanced by using microwave irradiation instead of conventional heating. In this study N-phenylbenzylamine was synthesized using bromobenzene and benzylamine with Cu catalyst. Different amino acids were used as ligands and the microwave irradiation was used to speed up the reaction.

Synthesis of N-phenylbenzylamine was carried out using a reaction mixture of, 0.1 mmol of CuI, 1.0 mmol of bromobenzene, 1.5 mmol of benzylamine, 1.5 mmol of dimethylsulfoxide (dimethylformamide and other solvent mixtures were also used), 1.0 mmol of potassium carbonate and 0.2 mmol of different amino acids. The reaction mixture with the particular amino acid was then heated in a microwave for one minute with 10 s intervals at different microwave powers (160 W - 800 W). Purification was carried out using liquid chromatography. The purified products were identified and characterized using NMR and IR spectroscopy and the resultant spectrum was compared with the reference spectra of N-phenylbenzylamine.

At the lowest microwave power (160 W), asparagine, methionine, tryptophan and proline gave more than 10% yield in one minute. At the highest microwave power (800 W) asparagine, cysteine, methionine, phenylalanine, tyrosine, tryptophan and proline gave more than 10% yield in 1 minute. The best yield was obtained with proline and it was 23.7%. The effect of microwaving time on the percentage yield of the above reaction was checked with selected amino acids. The increase in percentage yield with reaction time was observed when aspartic acid, methionine, tryptophan and proline were used as amino acids. Methionine and proline gave 36.3% and 36.9% yields respectively after 5 min at 160 W. The effect of solvents on the above reaction was checked with 80% DMSO in water, DMF and 80% DMF in water with selected amino acids. The results indicate that DMF is a better solvent for this reaction than DMSO.

Studies are underway to improve the yield of this reaction and once the conditions are optimized other nucleophilic aromatic substitution reactions will be tested.

Keywords: Ullmann condensation, N-phenylbenzylamine, amino acids, microwave radiation.