Methylation of 5-phenyltetrazole with dimethyl carbonate

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In recent years, dimethyl carbonate has gained an increasingly important role in the chemical industry, mainly due to its versatility as an environmentally friendly solvent and methylating agent. Dimethyl carbonate is suitable replacement for toxic dimethyl sulfate and methyl halides, which are widely used in organic chemistry [1]. Using 5-phenyltetrazole as an example, we have studied the possibilities of using dimethyl carbonate as a methylating reagent for NH-unsubstituted tetrazoles. It was found that heating of dimethyl carbonate solution of 5-phenyltetrazole under reflux does not lead to the formation of methylated products. When the reaction was carried out in boiling acetonitrile in the presence of K_2CO_3 , after 25 hours of the process a trace amounts of a mixture of 1- and 2-methyl-5-phenyltetrazoles was isolated (15 : 85). The best results are achieved when the process is carried out in dimethylformamide at 130 ° C in the presence of K_2CO_3 . As a result, a mixture of 1- and 2-methyl-5-phenyltetrazoles is formed with a yield of 90%, containing 13% of 1-methyl-5-phenyltetrazole and 87% of 2-methyl-5-phenyltetrazole.



The individual 1-methyl- and 2-methyl-5-phenyltetrazoles from the resulting mixture of isomers can be isolated by liquid column chromatography.

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References

[1] P. Tundo et al. Green Chemistry (2018) 1