Área: ORG **Nº de Inscrição: 00015**

Microwave-assisted multicomponent synthesis of julolidines using silica-supported calix[4]arene as heterogeneous catalyst

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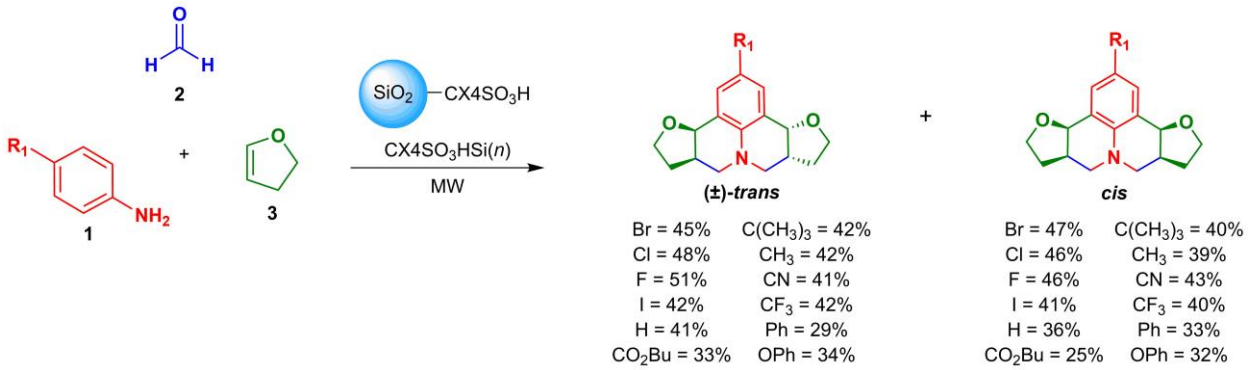
Palavras Chave: *Calix[n]arenes, Heterogeneous catalysis, Julolidines, Microwave-assisted, Multicomponent reactions.*

Highlights

Use of silica-supported calix[4]arene as a heterogeneous catalyst. Multicomponent synthesis of julolidines under solvent-free conditions and microwave-assistance. Recovery and reuse of the catalyst.

Abstract

In this work, the multicomponent Povarov reaction was used for the synthesis of tetrahydrofuranjulolidines catalyzed by Si(*n*)CX[4]SO_3H and assisted by microwave. The heterogeneous catalyst was synthesized from the *p*-sulfonic acid calix[4]arene by the sol-gel technique. The Povarov reaction is a multicomponent reaction between an aniline, an aldehyde and an alkene or alkyne, catalyzed by Lewis or Brønsted acids, and can provide tetrahydroquinolines, quinolines or julolidines as products, depending on the reactive conditions employed. In this work, 24 julolidines were synthesized from different *p*-substituted anilines, formaldehyde and 2,3-dihydrofuran. The optimized conditions for the synthesis were: aniline (0.5 mmol), formaldehyde (1.5 mmol), 2,3-dihydrofuran (1.5 mmol), temperature of 150 °C, reaction time of 10 minutes, catalyst concentration of 0.5 mol%, in the absence of solvents. Yields ranged from 58 to 97%. The julolidines were obtained as a mixture of diastereoisomers, which were separated by a silica gel chromatography column, providing 12 julolidines with *cis* stereochemistry and 12 julolidines with (±)-*trans* stereochemistry. Compounds were identified and characterized by mass spectrometry, infrared spectroscopy, chiral column HPLC and ^1H and ^{13}C Nuclear Magnetic Resonance. The great advantage of the developed methodology is the short reaction time, the operational simplicity and the absence of solvents, factors that help to reduce the formation of residues and go according to the principles of green chemistry.



Scheme 1. Scope of the Reaction for Different Anilines. Isolated yield.

References

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Acknowledgments

UFV, Capes, FAPEMIG, CNPq