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TEMPERATURE MEASUREMENTS IN MICROWAVE ASSISTED SOLVENT-FREE ORGANIC SYNTHESES

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The exact set up and measure of the reaction temperature is fundamental to establish the best reaction conditions for a particular synthesis. In solvent-free organic syntheses assisted by microwave irradiation, temperature control is one of the main parameters influencing the reaction behavior in terms of yield, product purity and reaction speed. Moreover the temperature profiles of the reactions can be considered a valid tool to compare the efficiency of microwave activation versus conventional heating under the same operative conditions (vessel, reaction time etc.).
In this work we present an investigation on a 1,3-dipolar intramolecular cycloaddition of tosylhydrazones derived from α,β-unsaturated carbonyl compounds with a hydrogen in β position, to obtain the relative pyrazole derivatives in solventless conditions [1].
The reactions were carried out using two monomode reactors: Synthewave™ 402 - Prolabo and Discover® CEM. The reaction temperatures were measured by infrared detectors pointed to the external wall of the reaction vessel and by optical fibers directly plunged in the reaction batch.
In the Prolabo oven the temperature was simultaneously measured by the IR sensor and by one or more external optical fibers (Neoptix Reflex, four channels) which were placed in different areas of the solid reaction medium.
As regards Discover® CEM, since the apparatus was equipped with its own optical fibers, there was the possibility to place the fiber in a desired position, but also to module the microwave forward power as a function of the temperature detected.
A comparison of the results is reported.

References

[1] Corradi, A., Grandi, R., Leonelli, C., Rizzuti, A., Rosa, R., Veronesi, P., Baldassari, S., Gambaro, R., Mariani, E., Villa, C., "Solvent Free Cyclisation of Tosylhydrazones of α,β-Unsaturated Carbonyl Compounds to Pyrazoles Under Microwave Irradiation", VIII Congresso INCA "Chimica sostenibile e Tecnologie ambientali: stato dell'arte e prospettive", Bologna, Italy, 23-24 March, 2006.