

TA009

ORGANIC CHEMISTRY

CAN MICROWAVE-ASSISTED REACTIONS AND PROCESSES BE QUALIFIED AND VALIDATED?

M. Nüchter^{1*}, B. Ondruschka¹, R. Bierbaum¹, D. Weiß², R. Beckert²

¹ Institute of Technical Chemistry and Environmental Chemistry,
Friedrich Schiller University of Jena, Lessingstr. 12, D-07743 Jena, Germany

² Institute of Organic Chemistry and Makromolekulare Chemistry,
Friedrich Schiller University of Jena, Lessingstr. 12, D-07743 Jena, Germany
E-Mail: matthias.nuechter@uni-jena.de

Since twenty years microwave irradiation is intensively used for the activation of chemical reactions and processes. A lot of these are favoured, if initiated by microwave irradiation instead a thermal (classical) energy source. A current, very extensive summary of microwave assisted synthesis results is given in [1] and a technology overview was published in [2].

Reaction mixtures adsorb microwave energy under determined substantial conditions and warm up from the interior to the system periphery (reactor wall) in contrast to conductive-convective heat transport. The reactions are often faster and in some cases more selective. The main precondition for the energy adsorption from the microwave field is the existence of polar (or polarizable) components in the reaction mixture.

We report here new insights in the energy efficiency of reactions in a microwave field and on the development of a concept to transfer thermal heated reactions to microwave assisted reactions by means of commercial, modular usable microwave set-ups. Particularly, questions of the qualification of technical microwave systems and of their measurement and monitoring technology, as well as of the validation of chemical reactions and processes are discussed. The following problems are inherent:

which system parameters influence the heating rate of substances in a microwave field,
do limits exist,
where are these limits located, and
why do these limits exist?

For that purpose, some technical microwave systems of various manufacturers were tested in order to determine their applicability to the implementation of chemical reactions and processes. Conversions and yields of some reactions are compared in dependence of irradiation modus, temperature and temperature measurement method. The power parameter of the various systems were determined externally and scrutinised, and conclusions are drawn for a possible scale up.

A draft for a comprehensive protocol (experiment description) is presented, which should guarantee the reproducibility of processes under microwave irradiation and which can thus facilitate the transfer to technical scale. Some examples of protocols will be presented from different fields of microwave application and different stages of scale up (synthesis, extraction, disinfection). At the end of the presentation the applicability of this method in student's education is presented [3]. The suitability of microwave energy in a new practical training in organic chemistry is demonstrated.

REFERENCES

- [1] P. Lidström, J. Tierney, B. Wathey, J. Westman, *Tetrahedron* **2001**, *57*, 9225-9283
- [2] M. Nüchter, B. Ondruschka, W. Bonrath, A. Gum, *Green Chemistry*, **2004**, *6*, 128-141
- [3] www.oc-praktikum.de