MICROWAVE PHOTOREACTOR FOR PHOTOCHEMICAL SYNTHESIS

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Introduction

Microwave chemistry is a very young scientific discipline and microwave (MW) irradiation represents a new way to increase the efficiency of many chemical processes. Microwaves can significantly increase both yield and selectivity of chemical reactions. Unique action of UV irradiation on a chemical reaction is very well recognized.
Our idea was to examine the effect of simultaneous MW and UV irradiation in the course of chemical reactions. For this purpose we have developed a simple laboratory MW photoreactor based on an electrodeless discharge microwave lamp (MWL). The MWL generates UV radiation when placed in a MW field. We have recently reported in papers [1-5] that a photochemical reactor consists of a MWL placed directly into a reaction mixture. For generation of a MW field we used a modified domestic MW oven. This simple arrangement allows simultaneous interaction of UV and MW irradiation with the studied photochemical system. This gives a unique possibility to study photochemical reactions, especially under extreme thermal conditions. It is well known that some photochemical reactions are sensitive to temperature, which may have a significant effect on the stereo- or regio-selectivity of the reaction. Moreover, since the MW field affects the dynamic behavior of the photochemically generated radical pairs, one can anticipate non-thermal influence of photochemical reactions in which such intermediates are involved. This paper describes our microwave photoreactor (made by simple modification of a domestic microwave oven) and its application in microwave photochemistry.

Microwave photoreactor

We have modified a commercially available MW oven (Whirlpool, model M 435) by 30 mm holes for a water cooler and a IR pyrometer or fiber optic thermometer (Nortech), according to Figure 1. A round part (200 mm diameter) of the oven bottom was replaced by an aluminium plate to allow magnetic stirring. The electrodeless mercury discharge lamp (MWL) was made from quartz or Pyrex tubings, filled with mercury and argon and sealed under reduced pressure of 20 mmHg. The lamp size varied from 10x20 to 20x40 mm. The contruction of MW photoreactor was described in more detail recently [5].

Typical experiment

Reaction mixture is placed into a reaction vessel (50-1000 ml) equipped with a stirring bar, along with the MWL (lamp may float or sink on the bottom). Such an arrangement allows direct irradiation of the entire volume of reaction mixture by UV light. The vessel is equipped with a very efficient water cooler. The temperature of the reaction mixture is monitored by the IR pyrometer or fiber optic thermometer. The MW photoreactor has been successfully tested in the addition reaction of tetrahydrofuran to carbon-carbon double bond and in the transformation of valerophenone.
The MW photoreactor also allows photocatalytic experiments when a heterogeneous catalyst for the photochemical reaction is used.

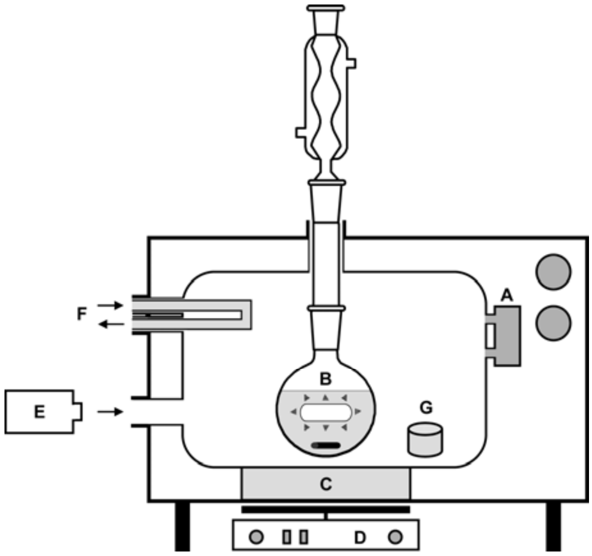

Fig.1. Domestic MW oven as MW photoreactor

Conclusion

The main advantage of our MW photoreactor is the possibility to carry out reactions under the simultaneous action of MW and UV irradiation while UV radiation is being generated by the MW field. Because most solvents and organic compounds are polar, they couple very strongly with microwaves and the reaction mixture can be superheated to cause further acceleration of reactions. The MW photoreactions can find applications in photochemical synthesis, in photocatalytic reactions and in processes like decontamination of water or decomposition of toxic compounds.

References

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