(86) The Microwave Photochemical Reactor for Synthesis

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ABSTRACT

The fact that electrodeless discharge lamp (or microwave lamp, MWL) generates ultraviolet (UV) radiation when placed into the microwave field has been known for long time. Low powered and low-pressure electrodeless lamps were utilized in spectroscopy and analytical chemistry four decades ago. However, its application for organic photochemistry has been shown for the first time by us in three subsequent publications. We reported on the studies in an original photochemical reactor consisting of MWL placed into the reactor vessel of a commercial microwave oven. The MW field generated UV discharge in the lamp with a consequence of the simultaneous UV and MW irradiation of the sample. Such simple arrangement brings a unique possibility to study photochemical reactions under extreme thermal conditions. In addition to our investigations, Chemat and his coworkers recently described a MW-UV reactor in which the UV source was external. In this work we wish to describe details of working with our MW-UV reactor, necessary modifications to a domestic microwave oven for microwave photochemistry experiments, and safety precautions. The paper reviews our current knowledge of this field as well as the industrial applications. Our modifications to a domestic microwave oven are similar to those described for microwave chemistry experiments. Several commercially available ovens were modified according to the Figure. In a typical design, at least four holes were drilled to the oven walls: two 30-mm holes, one for a condenser tube in the oven top and the other from the side for an IR pyrometer, and two 20-mm ports for a glass tube with circulating water (inlet and outlet). The opening for the IR pyrometer can serve for the external source of UV irradiation. The electrodeless discharge mercury lamps (MWL) were made of quartz or Simax (Pyrex equivalent) tubings, filled with mercury and argon, and sealed under 20-Torr vacuum. Quartz lamps are suitable for irradiations at wavelengths longer than 254 nm, while Simax absorbs most of UV irradiation below 280 nm. The size of our lamps varied from 10x20 to 20x50 mm but this size range is not limiting. The spectral characteristics of the electrodeless lamp resemble more those of high-pressure mercury lamps and are known. The lamp gives over three times as much UV radiation as the conventional electrode lamp. The typical experiment consisted of a reaction vessel (25-1000 ml) with a reaction mixture, equipped with a Teflon-covered stir bar and MWL (larger lamps usually floated in the liquids while smaller ones sank). Such arrangement allowed irradiating the whole volume of the solution by UV light. The vessel was connected to a very efficient water-cooled condenser by a 100-300 mm long glass tube. The microwave power was adjusted to a maximal value of the particular oven (800-900 W), which guaranteed a continual MW radiation. The circulating cool water or some amounts of a solid, MW absorbing, material (basic Al_2O_3, molecular sieve etc.) were used in those cases when a small quantity (<50 ml) of a polar liquid or non-absorbing (non-polar) liquids for microwave photochemical experiments were used. It removed the excess microwave power and/or prevented the magnetron to be destroyed by overheating. Since MW absorbers may lower the MW power of the reactor the same amount of the material is essential for a good experimental reproducibility. The temperature of a liquid was monitored an IR pyrometer or by a fiber-optic probe. The lamp was initiated in non-polar solutions usually in several seconds when it warmed up. Each liquid started to boil very quickly because of the lamp heat operation. Polar liquids (e.g., water, ethanol, or acetonitrile), when used in higher amounts, often prevented the lamp initiation because they absorb most of the microwave power. We recently described a number of photochemical systems that were studied in our microwave photochemical reactor. The reactor has been studied in terms of an operating MW power and MWL heating capabilities, or the MWL quality and the scale of the experiment. In addition, efficiencies of several photochemical experiments under MW conditions were compared to those using a conventional UV lamp. The advantages and disadvantages of MWL application has been formulated - the electrodeless lamp was presented as a very simple, economic, and efficient tool for photochemistry.