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Domestic Microwave Ovens in the Laboratory

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Microwaves provide the only method of heating that does not use thermal conduction. While infrared is absorbed on the surface of the material, microwaves penetrate several centimeters deep and dissipate the energy they carry to the heart of the material. The only condition is that the material must not be a good electrical conductor.

Microwaves activate polar molecules, especially water. Water heats by absorption of microwaves and evaporates, creating local high pressures, which make the water migrate from within the heart of the object to the surface. Drying is fast and homogeneous, because the dry parts do not absorb as much energy as the wet parts and do not heat up excessively. Thus, an ordinary microwave oven, designed for domestic use, can have many applications in the laboratory. We have used the Toshiba ER-7620, which delivers 650 W according to the manufacturer, for several purposes, mainly drying and regeneration of materials.

Using the Microwave in the Laboratory

Drying of Glassware. Glassware for use in experiments involving water-sensitive compounds, such as organolithium, organocuprate compounds, and iminium salts, can be dried rapidly and efficiently by microwave heating.[1] The students can dry glassware in minutes instead of having it prepared by the assistant. Drying of a complete distillation set only needs 10–15 min at full power.

Activation of TLC Plates, on Glass or Plastic. When preparing preparative TLC plates coated with an adsorbent such as Merck's Kieselgel HF 254, it is convenient to activate the plate by microwave heating. For a 20- × 10-cm plate, 15 min heating on half power is enough, and the plate cools very rapidly. This activation gives the same result as two hours in a 100 °C furnace.

Development of TLC Plates. Plastic TLC plates can be easily developed by spraying concentrated sulfuric acid on the plate, followed by irradiation for 30–60 s at full power. Most products give a beautiful set of different colors by slight decomposition. There is no risk of melting the plate, as the material it is composed of does not absorb microwaves.

Regeneration of Chromatographic Adsorbent. The silica used in the laboratory, after washing with the appropriate polar solvent (e.g., methanol), can be dried by microwave heating and then reused. The particularly expensive silica used for flash chromatography (e.g., Merck 9385 Silica gel 230–400 mesh) according to Still[2] can thus be reused after regeneration. Microwaving offers the possibility of washing the silica with water, to desorb very polar products, and then of desorbing the water with microwave energy.

Regeneration of Drying Material. Self-indicating granulated silica gel, used as drying material, can be easily reactivated by microwave heating when saturated with water. For example, 20 g of silica gel saturated with 6 g of water were totally regenerated by 5 min at full power .

Regeneration of Molecular Sieves. Volatile liquid compounds molecularly adsorbed within porous granules of a molecular-sieve adsorbent can be recovered by microwave heating. When regenerating adsorbent in a column, the pressure generated by the evaporation promotes the discharge of the condensed liquid from the bottom of the column, without the assistance of a carrier gas or the application of reduced pressure.[3] To accomplish this, the column must be kept vertical. Small quantities of adsorbent can be regenerated in any open vessel, if the adsorbent layer is not too thick. In the dehydration of zeolites, the molecules are desorbed directly by the microwave field, the process being independent of the temperature of the solid.[4] It has been reported that some zeolites melt in seconds under microwave irradiation.[5]

Precautions and Problems

The essential problems one can have while using a commercial oven are caused by nonhomogeneous distribution of the energy. High energy focused on a narrow spot may cause the breakage of TLC glass plates and not allow reproducible plate activation. The following method for determining energy distribution is inexpensive and easily carried out. A filter paper sheet, the size of the inner oven, is impregnated with a solution of 0.5 g cobalt chloride in 10 mL water. It is left to dry on a flat surface, and then turned back to pink over a hot water bath. It is irradiated in the oven on low power. The irradiation should be stopped as soon as blue zones appear on the paper sheet. It is then possible to determine if heating in the oven is homogeneous, or whether a turntable is needed. Many ovens are now sold with a built-in turntable, and the purchase of such devices is recommended.

Precautions must be taken for the evaporation of organic solvents in a microwave oven: An electric spark is always possible between any metallic particle and the nearest part of the oven, causing the ignition of the vapors and the explosion of the oven. If microwaves are used to regenerate chromatographic adsorbent, the Silica gel should be predried by flushing the column with air to evaporate excessive quantities of flammable solvents such as methanol. Any microwave oven used in the laboratory should be placed under an efficient hood. Metallic particles inside the oven must be avoided, and no closed vessel should be used, except those specially designed for high pressures, generally made from Teflon.[6]

[1]Hasek, J. A.; Wilson, R. C. *Anal. Chem.* **1975**, *46*, 1160.
[2]Still, W.C.; Kahn, M.; Mitra, A. *J. Org. Chem.* **1978**, *43*, 2923.
[3]Burkholder, H. R.; Fauslow, G. E. U.S. patent US 4,421,651; 20.12.1983.
[4]Roussy, G.: Zoulalian, A.; Charreyre, M.; Thiebaut, J. M. *J. Phys. Chem.* **1984**, *88*, 5702.
[5]Komarneni, S.; Roy, R. *Mater. Lett.* **1986**, *4*, 107.
[6]Such devices can be bought at CEM Corporation, Post Office Box 200, Matthews, North Carolina 28106, (704)821-9204, 1-800-334-6317, or at Berghof Gmbh, Abt. Labortechnik, Harretstrabe 1, D-7412 Eningen, West-Germany.