

MICROWAVE PHOTOCHEMISTRY



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MICROWAVE PHOTOCHEMISTRY

= combination of **UV/VIS** and **microwave** irradiation



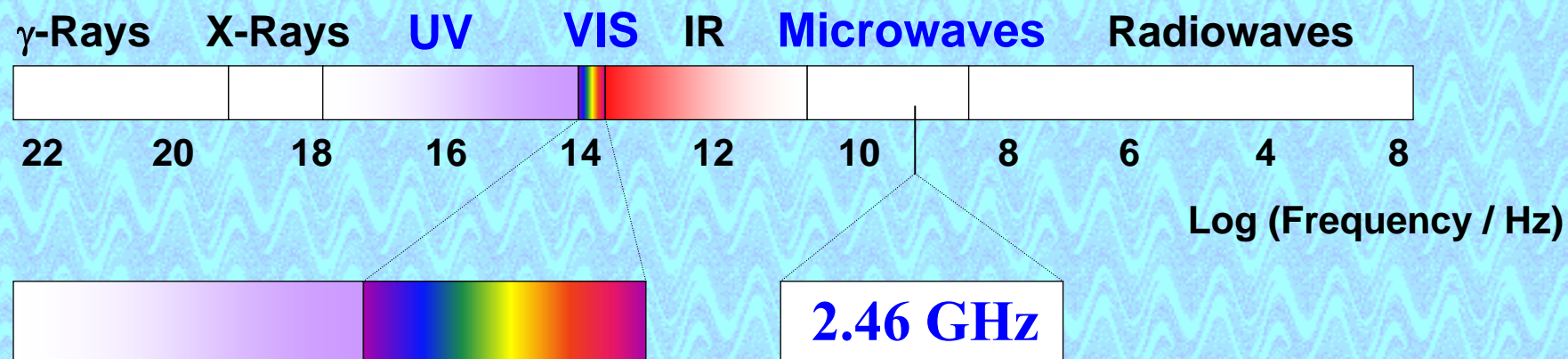
$\lambda \approx 180 - 700 \text{ nm}$

$E \approx 660 - 170 \text{ kJ/mol}$

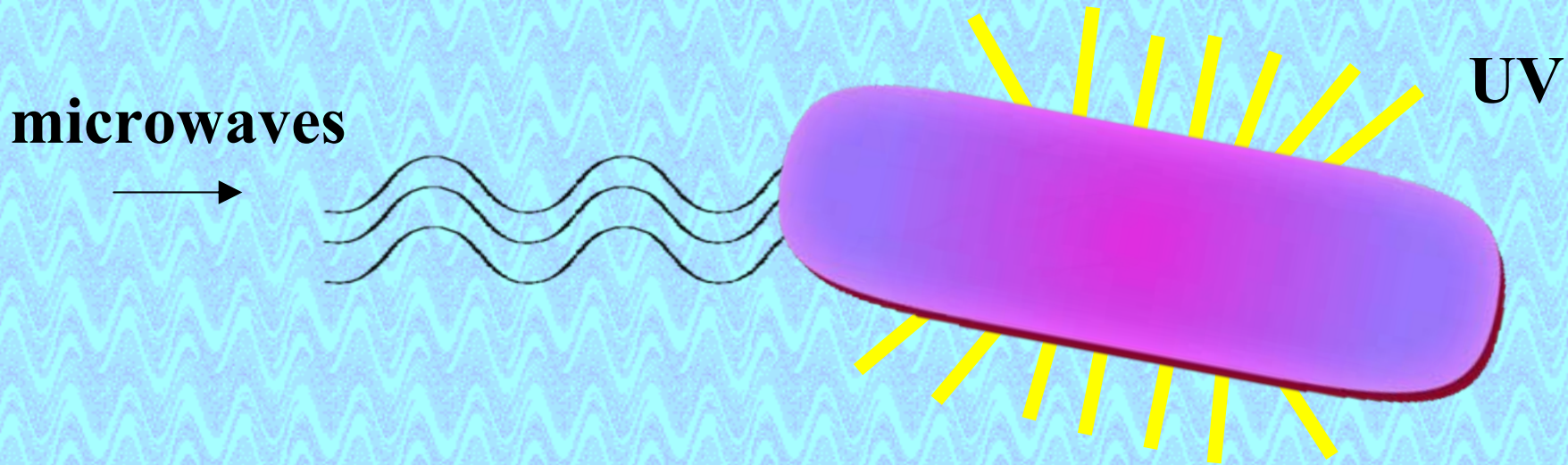


$\lambda \approx 12 \text{ cm}$

$E \approx 1 \text{ J/mol}$



Electrodeless Discharge Lamp (EDL)



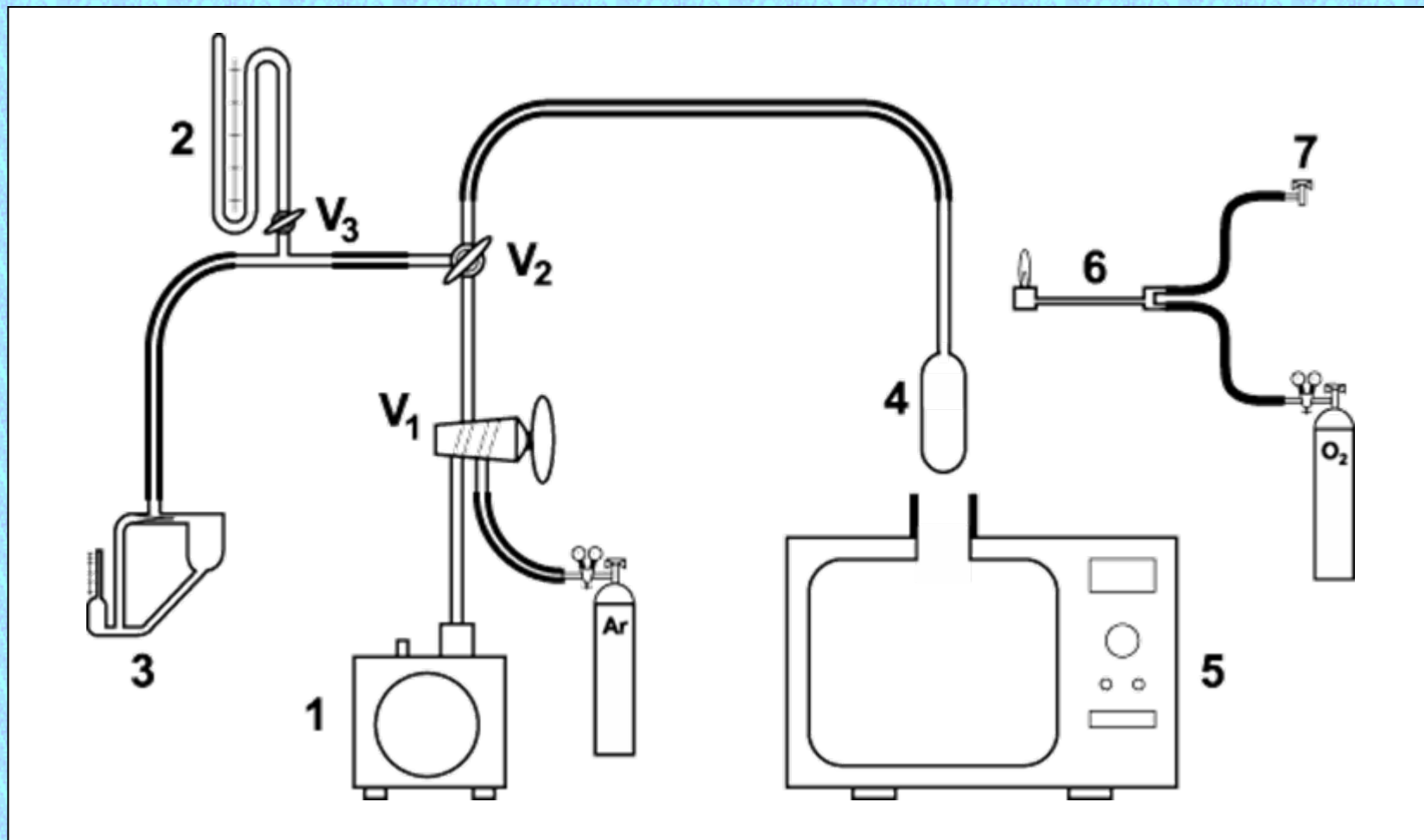
- ◆ glass envelope (20 x 40 mm)
- ◆ filling material (1-10 mg)
(Hg, Cd, P, I₂, S, Se)
- ◆ argon (5 Torr)



Principle of Hg-EDL operation



Preparation of EDL

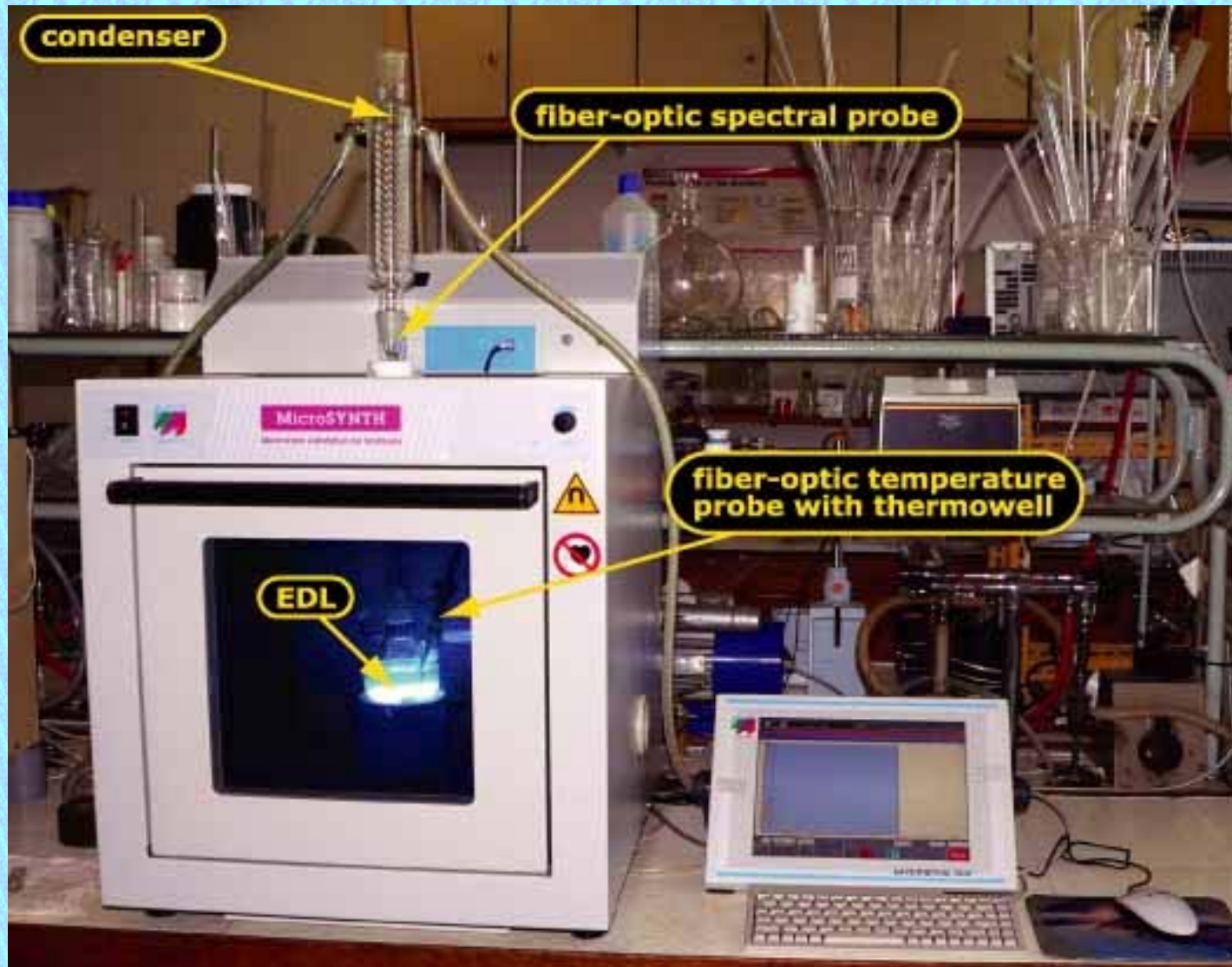


1 - rotary oil vacuum pump
2 - Hg manometer (0-200 Torr)
3 - McLeod gauge (0.01-10 Torr)

4 - EDL blank (Pyrex)
5 - modified MW oven
6 - glass-working burner

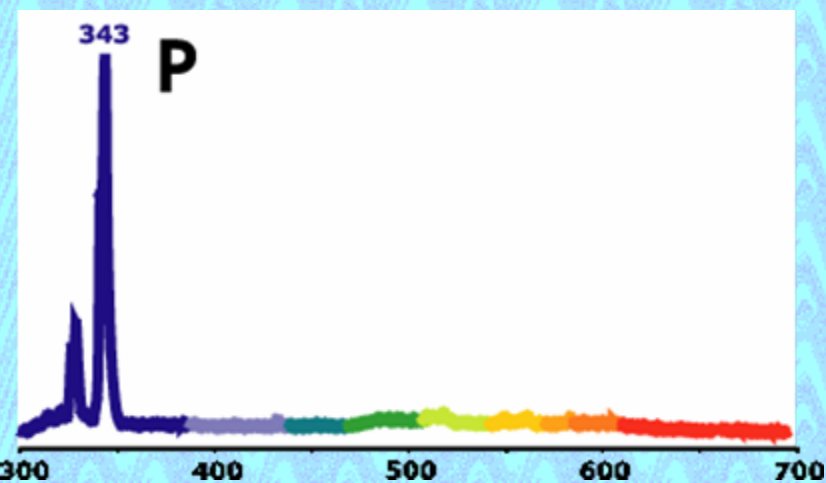
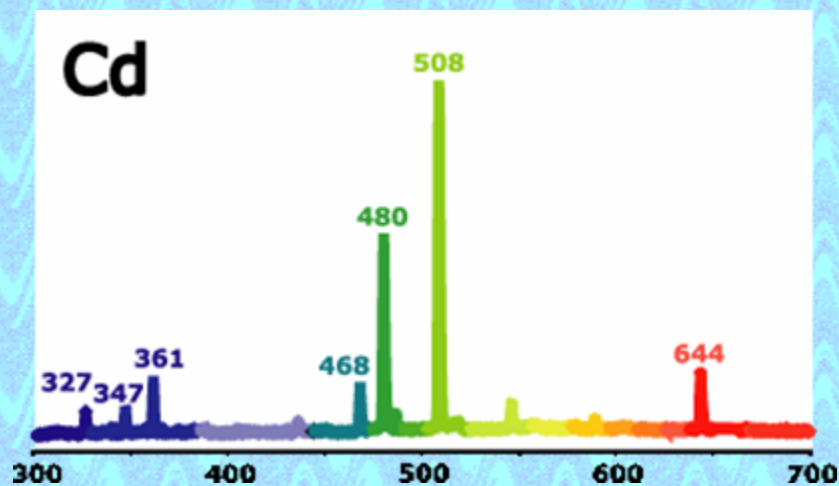
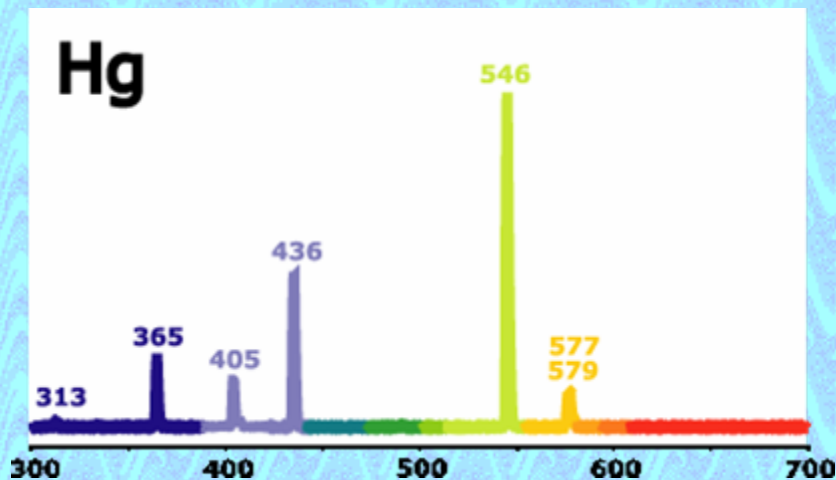
7 - natural gas
silicone rubber
hoses

Testing of EDL Performance for spectral measurements (MicroSYNTH, Milestone)



Spectral Characteristics of Pyrex-EDLs

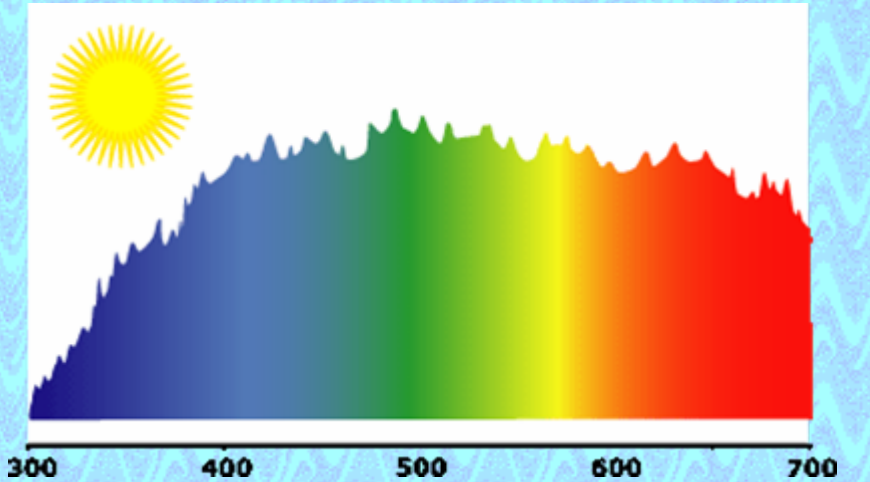
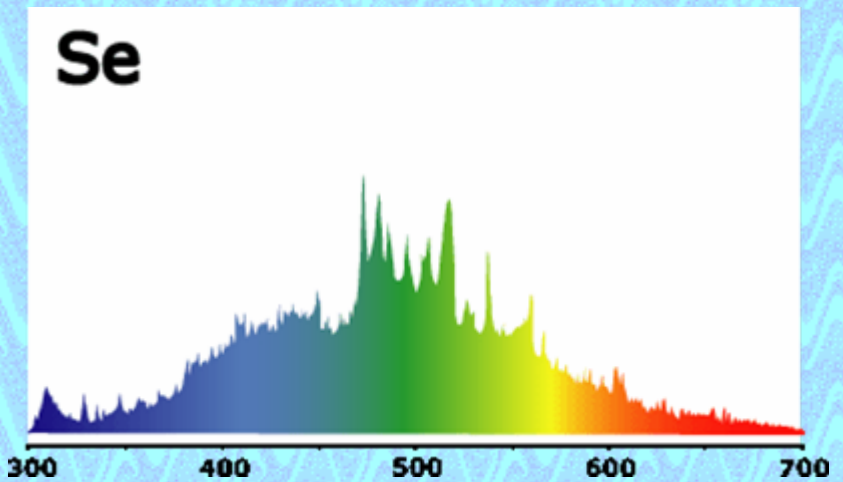
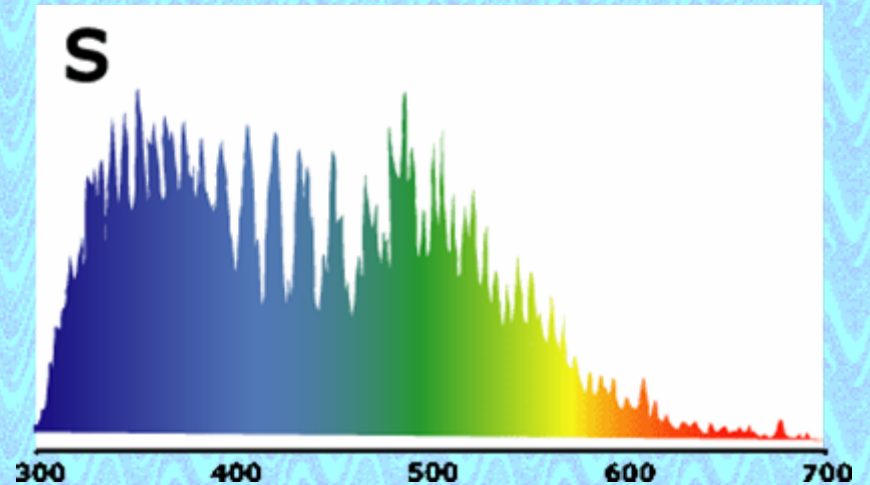
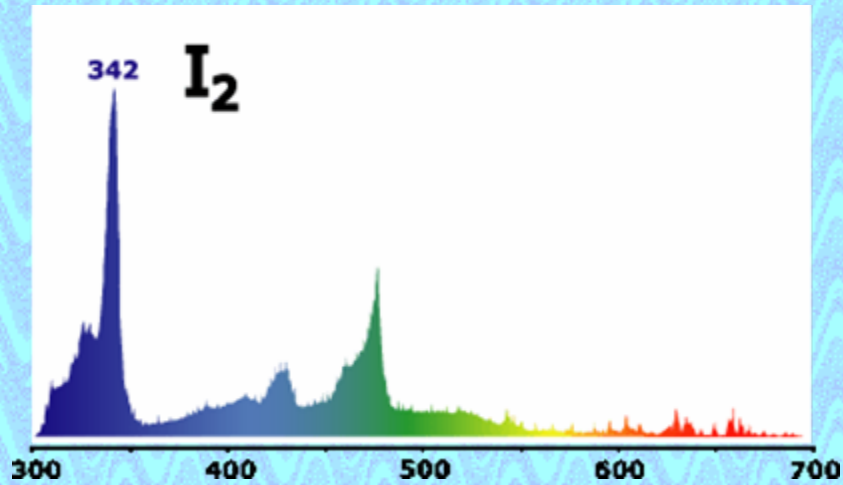
Line emission spectra



[P. Müller, P. Klán, V. Církva, J. Photochem. Photobiol. A: Chem. *158* (2003) 1]

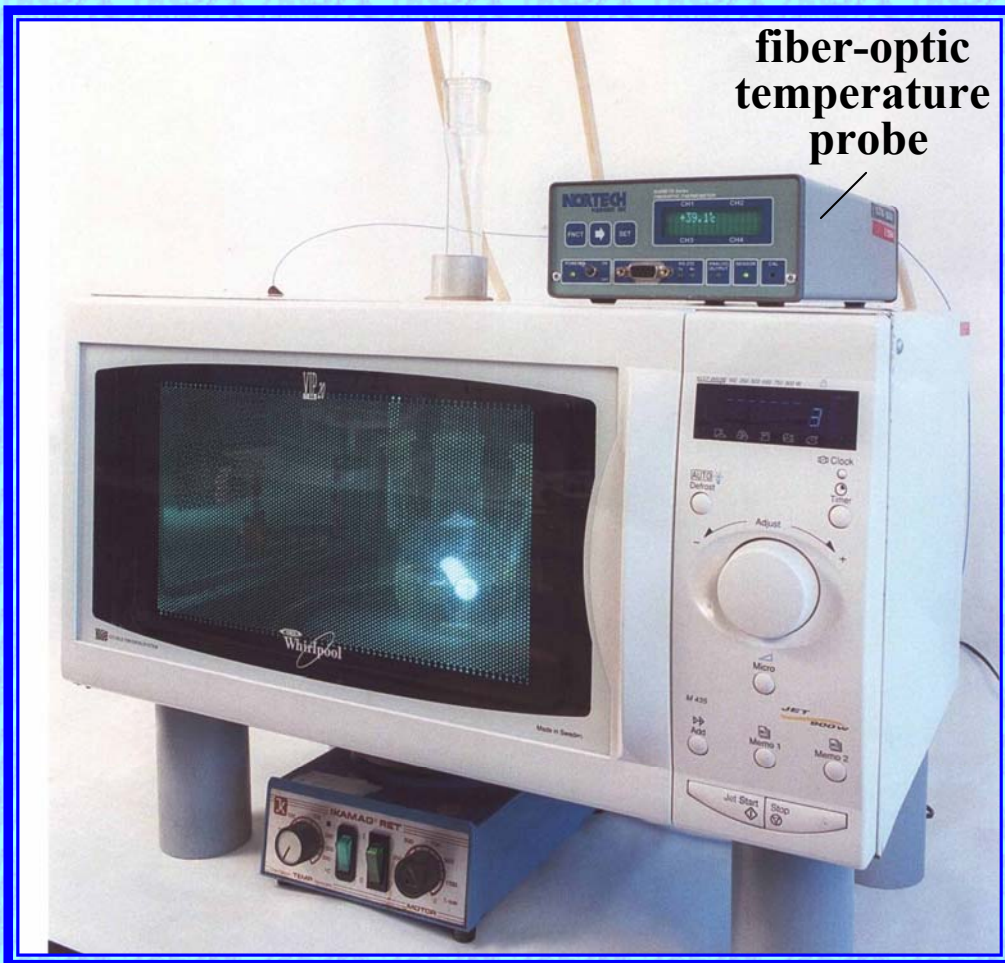
[P. Müller, P. Klán, V. Církva, J. Photochem. Photobiol. A: Chem. *171* (2005) 51]

Continuous emission spectra



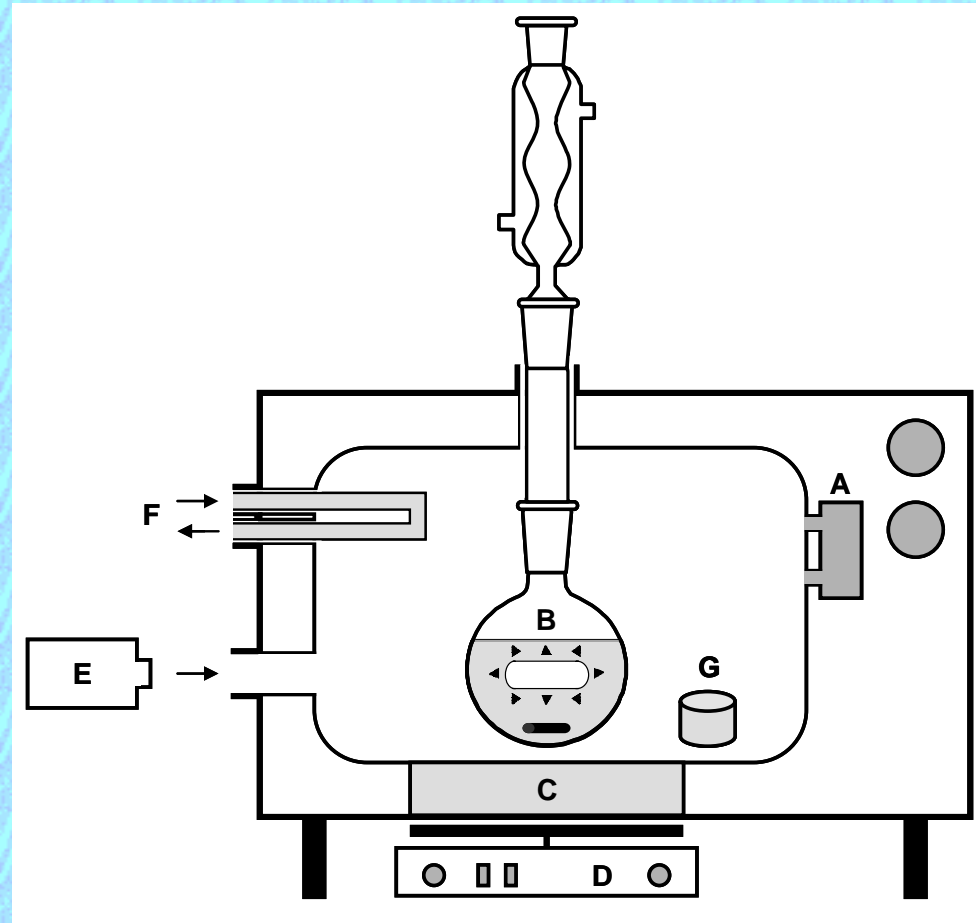
[P. Müller, P. Klán, V. Církva, J. Photochem. Photobiol. A: Chem. *171* (2005) 51]

Photochemical Applications of Pyrex-EDLs



fiber-optic
temperature
probe

**Batch microwave
photoreactor**



A – magnetron

B – flask with EDL

C – aluminum plate

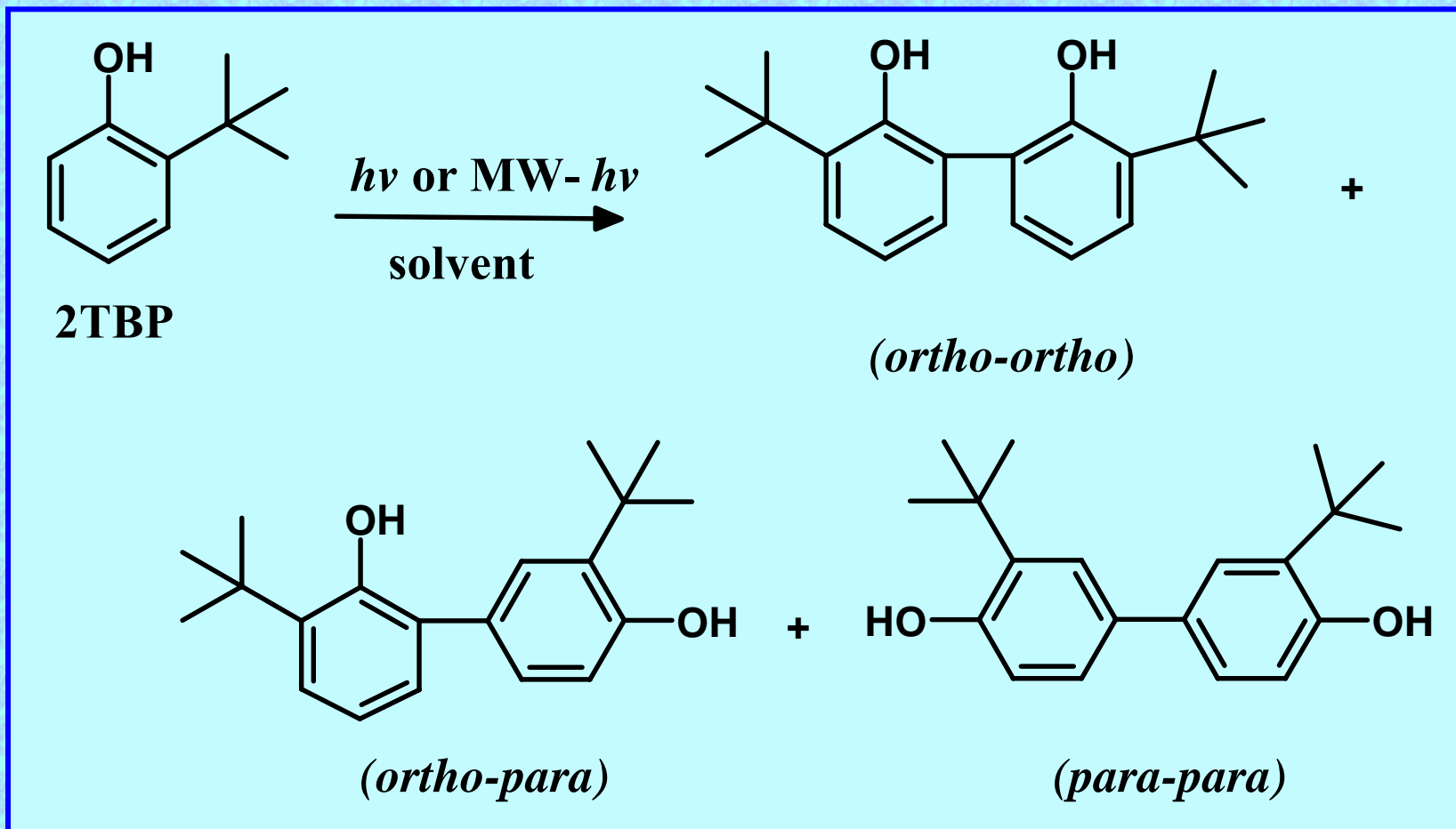
D – magnetic stirrer

E – IR pyrometer

F – circulating water

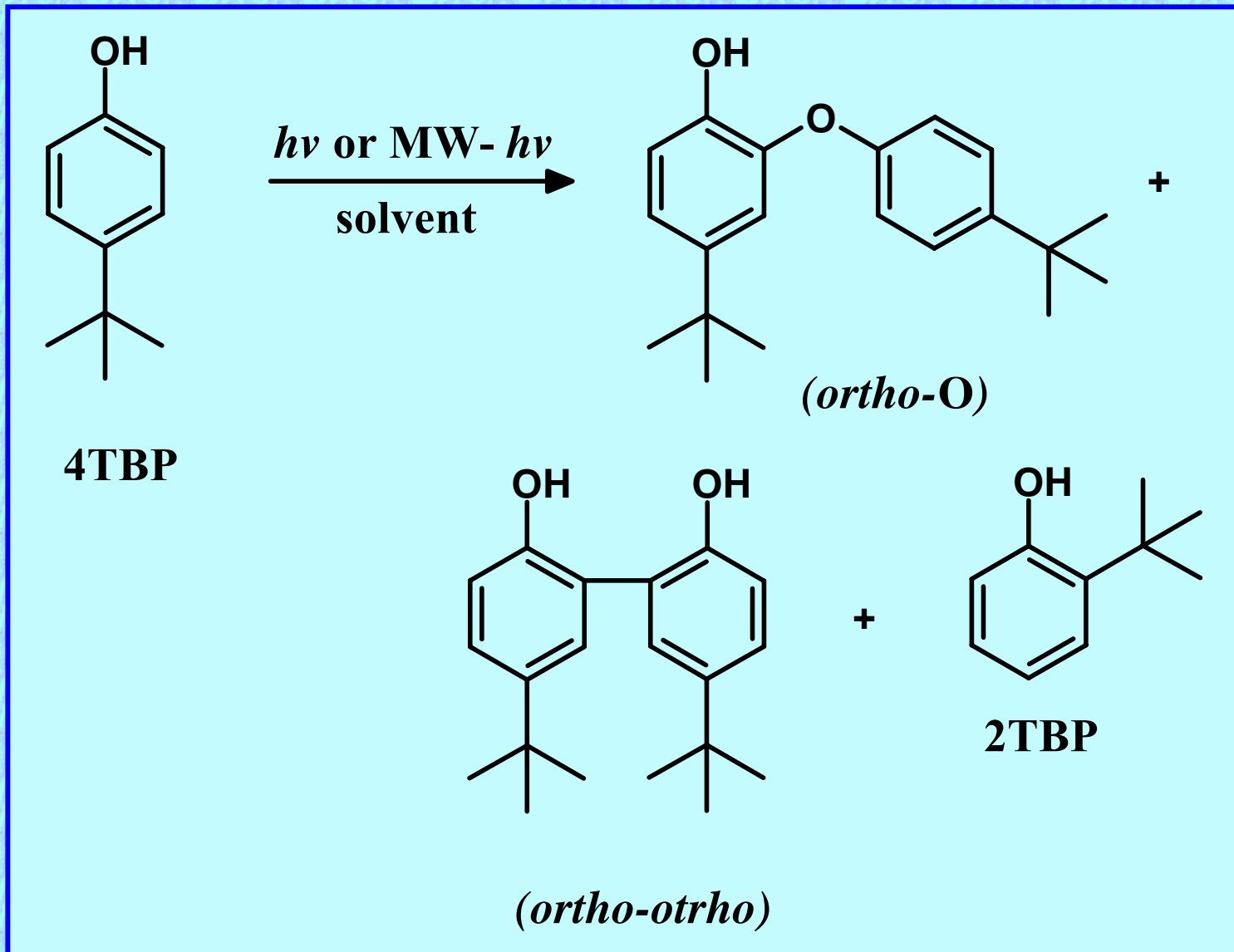
G – dummy load

Transformation of 2-*tert*-butylphenol



[V. Círka, J. Kurfürstová, J. Karban, M. Hájek, J. Photochem. Photobiol. A: 168 (2004) 197]

Transformation of 4-*tert*-butylphenol



Advantages

- 1) Simultaneous MW and UV/VIS irradiation of the sample at higher temperature (at reflux)**
- 2) Use of commercially available or inexpensive domestic MW oven**
- 3) Simplicity and accessibility of the experimental set-up and a low cost of EDL**
- 4) Photochemical efficiency of EDL in modified MW oven and easy method of EDL preparation in lab**
- 5) Knowledges of EDL spectral characteristics are essential for design of photochemical experiments**
- 6) Pyrex-EDL absorbs most of the UV below 290 nm without the necessity of filtering off the undesirable part of radiation**

**Klán P., Círka V.
(A. Loupy Ed.)
Chapter 14
Microwave Photochemistry
463-486 pp**

