

## Raztapljanje vzorcev (II)

Zaprti sistemi, uporaba mikrovalov, alternativni pristopi

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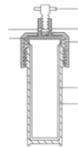
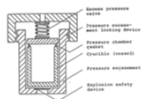
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## Priprave za razkroj vzorcev v zaprtih sistemih (avtoklavi)



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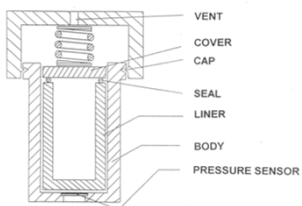
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## „Klasični“ avtoklav za razkroj vzorcev pri povišanem tlaku

“Classical” system for pressurized decomposition



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### Spekter elektromagnetnega valovanja

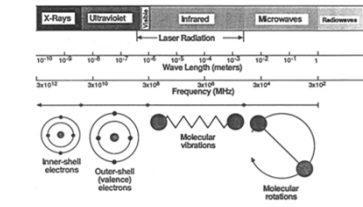


Figure 2.1. Electromagnetic spectrum.

MW  
 915 ± 25 MHz  
 2450 ± 43 MHz  
 5800 ± 75 MHz  
 600 - 700 W

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### Interakcije mikrovalov s snovjo

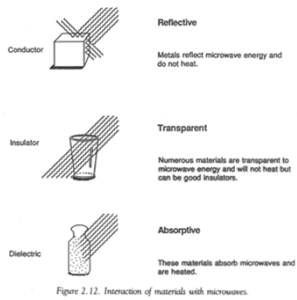


Figure 2.12. Interaction of materials with microwaves.

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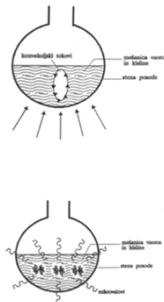
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### Princip segrevanja z mikrovalovi




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Globine prodiranja mikrovalov v vodi (odvisnost od frekvence elektromagnetnega valovanja)

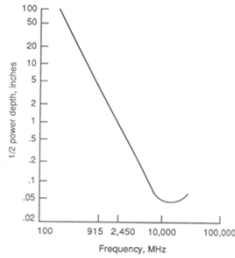


Figure 2.3. Variation of penetration with frequency for water at 25 °C. (Reproduced with permission from ref. 13. Copyright 1975 AVI Publishing).

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Delež absorbirane moči mikrovalov, glede na maso

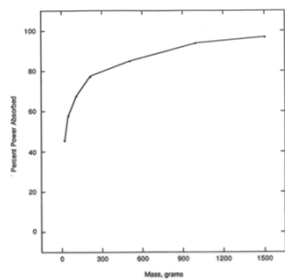


Figure 2.4. Percent microwave power absorbed by water. (Reproduced with permission from ref. 21.)

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Sposobnost različnih materialov za absorpcijo mikrovalov

Tabela 1: Faktorji tan δ za različne materiale\*

Material	Temperatura [°C]	Tan <sup>δ</sup> (x10)
Voda	25	1570,0
Kremen	25	0,6
Keramika F-66	25	5,5
Porcelan št. 4462	25	11,0
Fosfatno steklo	25	46,0
Borosilikatno steklo	25	10,6
Corning steklo št. 0080	25	126,0
Pleksi steklo	27	57,0
Najlon 66	25	128,0
Polivinil klorid	20	55,0
Polietilen	25	3,1
Polistiren	25	3,3
Teflon PFA	25	1,5

\* Meritve pri 3000 MHz.

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Tabela 2: Moč, ki jo absorbirajo različne kisline<sup>4</sup>

Reagenti in koncentracija	50 ml	100 ml	200 ml
H <sub>2</sub> O	344±9	408±3	468±5
HNO <sub>3</sub> (16 M)	184±2	234±5	313±4
HNO <sub>3</sub> (1 M)	212±3	269±6	332±3
HF (29 M)	167±3	238±12	315±15
H <sub>2</sub> SO <sub>4</sub> (18 M)	231±6	331±4	396±8
HCl (12 M)	148±3	173±6	251±2
HCl (6 M)	138±3	190±4	253±4
HCl (1 M)	227±4	287±7	340±5

Moči so izražene v W, n=5

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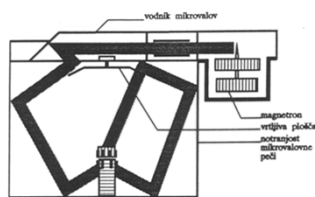
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### Shema mikrovalovne pečice

Slika 4: Shema mikrovalovne pečice<sup>4</sup>




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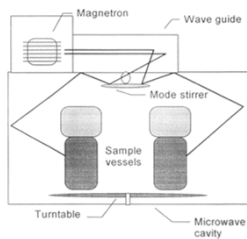
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### Laboratorijska mikrovalovna pečica

Microwave assisted decomposition:  
normal temperature/turntable system




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### Laboratorijska mikrovalovna pečica z avtoklavi

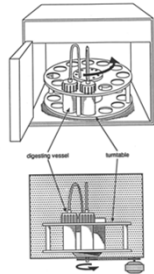


Figure 2.15. Schematic of a rotatable system used for heating digestion vessels in the microwave cavity

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### Segrevanje platinskih lončkov v mikrovalovni pečici

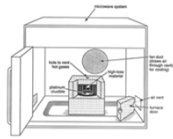


Figure 2.16. Schematic of a microwave single furnace

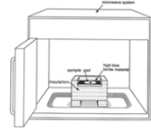


Figure 2.17. Schematic of a microwave alloy block

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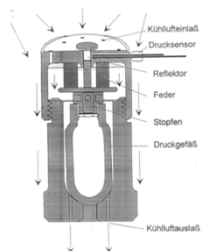
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### Sistem za mikrovalovni razkroj (posamezni avtoklavi)

Microwave assisted decomposition:  
PMD system for higher temperatures



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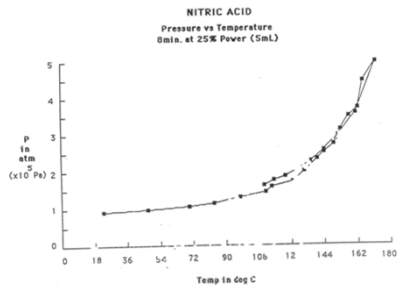
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Odvisnost tlaka od temperature v avtoklavu  
(dušikova(V) kislina)




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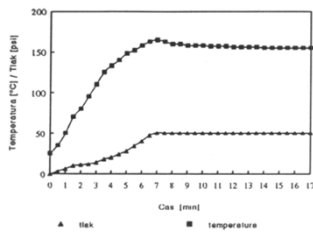
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Zveza med tlakom in temperatu




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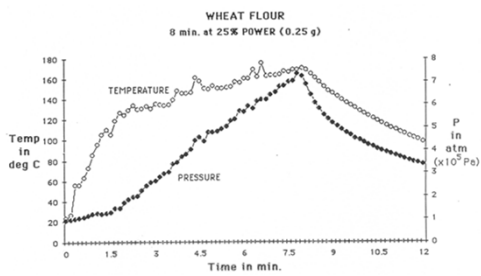
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Razkroj pšenične moke pri povišanem tlaku




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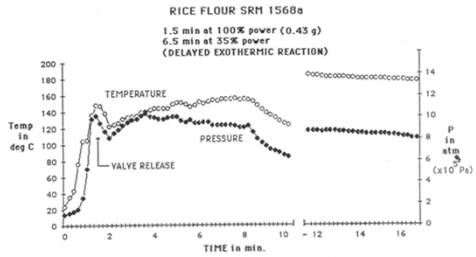
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Razkroj organskega materiala (riževa moka) – prikaz eksotermnosti reakcije




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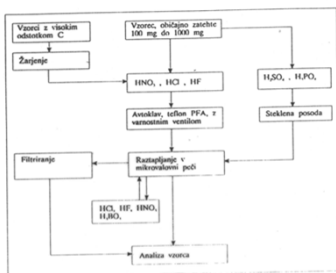
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Priprava vzorca z mikrovalovi

Slika 8: Stopnje raztapljanja v mikrovalovni peči




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Priprava vzorcev z mikrovalovi- metalurški vzorci

Tabela 3: Postopki za raztapljanje metalurških vzorcev v mikrovalovni peči

Vzorec	Zatehta	Volumen kislin [ml]			V[ml] H <sub>2</sub> O	Stopnja	Čas [min]	Moč [W]
		HCl	HNO <sub>3</sub>	HF				
n.jeklo**	100-500	3	3	3	1	5	100	
Al zlitine s Si <sup>22</sup>	200	9	3	2	1	2	100	
					2	10	35	
					3	3	100	
Fe-Mn- žindre <sup>22</sup>	500	10		2	1	0,75	100	
					2	30	30	
					3	1,75	100	
					4	10	40	
Cu zlitina <sup>22</sup>	400	10	5		1	15	60	
					2	5	0	

\* n. jeklo- nerjavče jeklo

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Steklena posoda za razkroj pri normalnem tlaku v mikrovalovni pečici

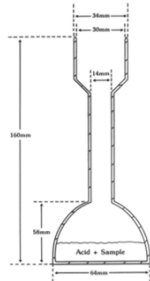


Figure 4.1. Open (Koblenz) glass vessel.

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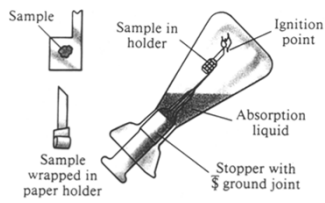
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Klasičen sežig organskih vzorcev v kisiku




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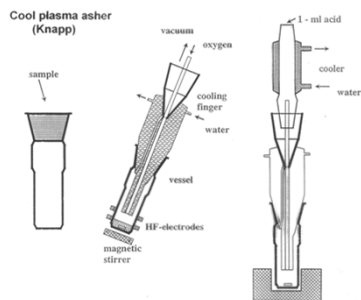
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„Hladni“ sežig v plazmi (po Knappu)




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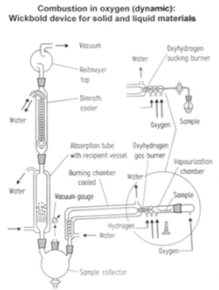
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Dinamični razkroj (sežig) v kisiku; tekoči in trdni vzorci




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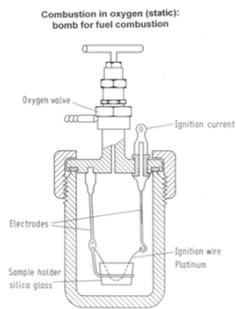
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Priprava (avtoklav) za sežig vzorcev z organsko osnovo v kisikovi atmosferi




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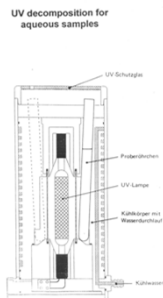
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Priprava za UV razkroj tekočih vzorcev




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