# Microwave basic techniques compared to its new revolutionary single reaction chamber (SRC) system

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**17 July 2013**

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# Introduction of Microwave

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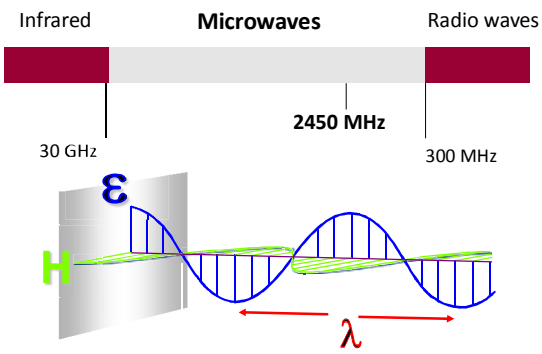
## Microwaves




ε : Electric field
H : Magnetic field
λ : Wavelength (12,2 cm for 2450 MHz)

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# Microwaves

- Microwaves are electromagnetic energy
- Microwaves frequency ranges from 300 to 300.000 MHz
- Microwaves wavelength ranges from 1 m to 1 mm
- Frequencies for industrial, medical and scientific uses are 915 MHz, 2.450 MHz (12,25 cm wavelength), 5.800 MHz and 22.125 MHz

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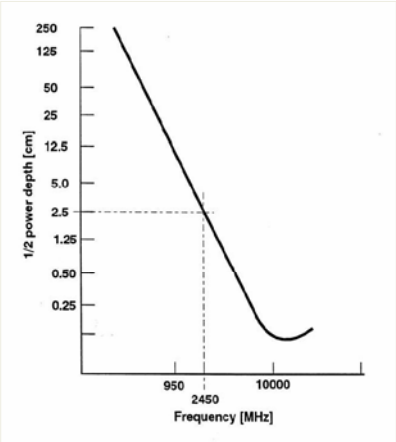
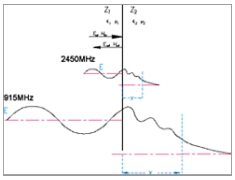
# Microwave penetration vs. frequency (water at 25°C)

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**Penetration depths of microwave energy of various materials at 2450MHz**

| Material | Temperature in °C | Penetration depth in cm |
|---|---|---|
| ice | -12 | 1100 |
| bread | 25 | 2 ... 5 |
| potato , raw | 25 | 0,9 |
| mashed potato | 25 | 0,8 |
| peas, carrots | 25 | 1 |
| meat | 25 | 0,9 ... 1,2 |
| paper, cardboard | 25 | 20 ... 60 |
| wood | 25 | 8 ... 350 |
| hollow glas | 25 | 35 |
| porcelain | 25 | 56 |
| polyvinylchloride20 | 20 | 210 |
| epoxy resin (Araldite CN-501) | 25 | 4100 |
| teflon | 25 | 9200 |
| quartz glas | 25 | 16000 |

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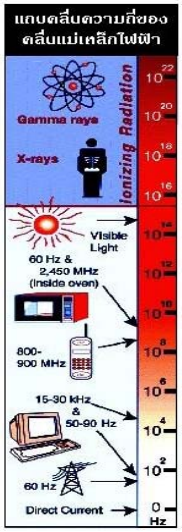
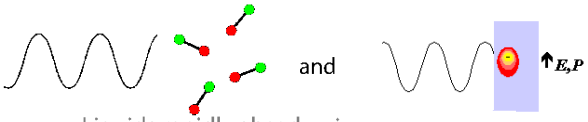
# Electromagnetic Energy Frequency ranges




- Liquids rapidly absorb microwave energy

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# Microwave energy

- Microwaves are not ionizing radiations
- Microwave energy is largely below the energy necessary to break the bonds of common organic molecules

| Microwave radiation (at 2.450 MHz) quantum energy (eV) | 0,0016 |
|---|---|
| **Chemical bond energy (eV)** | |
| H-OH | 5,2 |
| CH3-CH3 | 3,8 |
| Hydrogen bond (water) | 0,21 |

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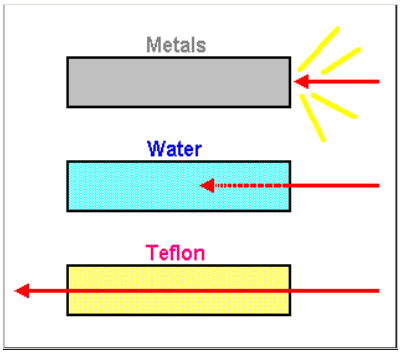
# Interaction of materials with microwaves




- Materials may be reflective, absorptive or transparent to microwaves

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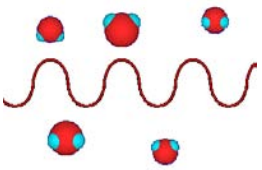
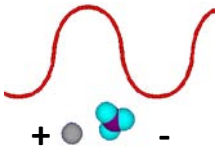
# Microwave Heating Mechanism

**Dipole Rotation**



**Ionic Conduction**




Alignment with the oscillating electric field
Loss of energy in form of heat

Mingos, D. M. P. *et al.*, Chem. Soc. Rev. **1991**, 20, 1 and **1998**, 27, 213

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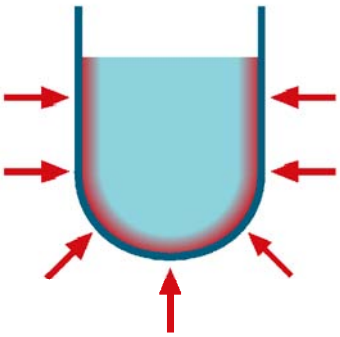
# Conductive Heating



- Heat passes through the vessel walls prior reaching the reactants
- Slow and inefficient method dependent from vessel material thermal conductivity
- Vessel temperature in excess of reaction mixture temperature

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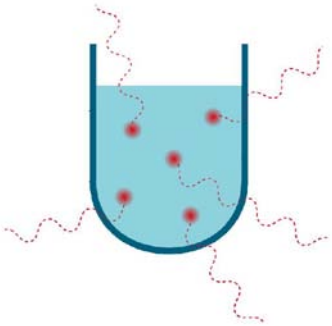
# Microwave Heating



- Microwaves couple with reaction mixture molecules
- Rapid temperature increase
- Independent from vessel material thermal conductivity
- Instantaneous localized super-heating by ionic conduction and dipole rotation
- No inertia (instant on-off)
- Dependant from ionic conduction and dipolar polarization

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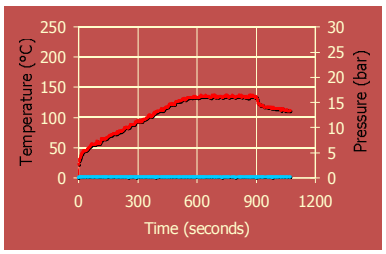
# Open vessel microwave heating

- 10mL of $HNO_3$ are heated in an open Teflon TFM vessel at 300Watt for 15'
- The highest temperature is just above the $HNO_3$ boiling point (125°C)
- Heating rate doubles when double power is applied

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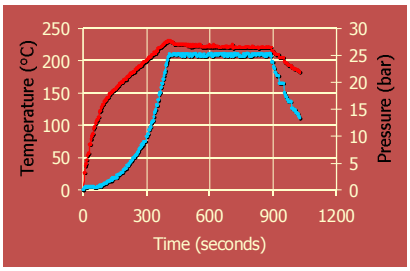
# Closed vessel microwave heating

- 10mL of $HNO_3$ are heated in 6 Teflon TFM closed vessels at 600Watt for 15 minutes
- The vapor pressure generated inside the vessels increases the boiling temperature of $HNO_3$

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# Pressure and Temperature

- Pressure is the mean and temperature is the goal
- Increasing the temperature by 10°C doubles the rate of a reaction
- Rapid microwave heating and the use of closed vessel allow for reducing the sample preparation time from hours to minutes

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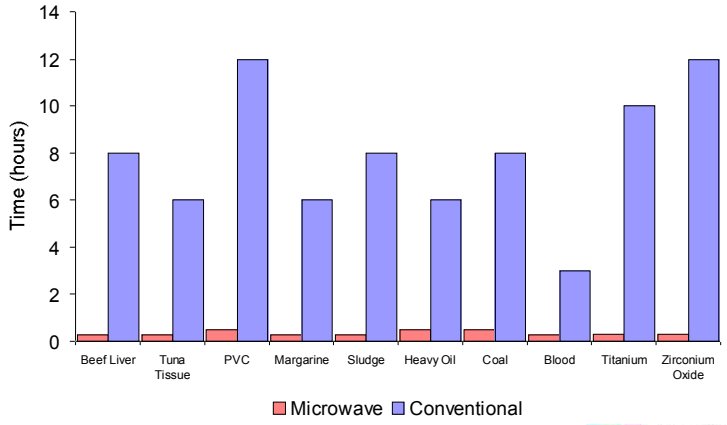
# Sample Preparation Time

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# Sample Preparation Quality

- No losses of volatile elements, complete recovery of Hg, Se, As etc.
- Low blanks, minimum quantities of acids are used
- No sample contamination from the environment or from other samples
- Reproducible and fully documented sample preparation procedure
- No acid fumes for improved laboratory personnel and working conditions

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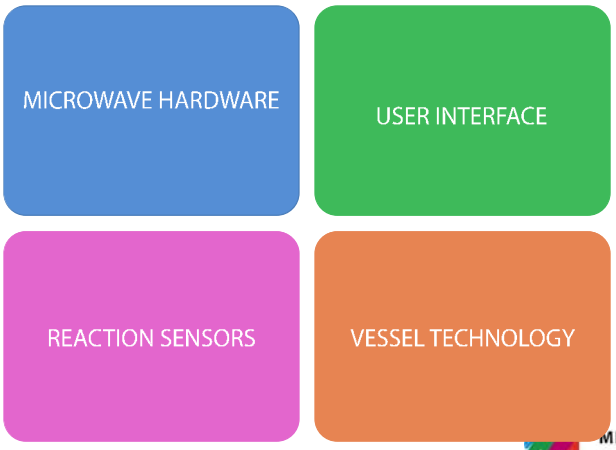
# A microwave system is the combination of:

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# Milestone ETHOS One

The best choice
in microwave sample preparation


## MICROWAVE HARDWARE

- Highest microwave power
  - Dual 900 Watt-rated magnetrons
  - Diffuser
- Pressure-responsive door
- Door locking system
- SafeVIEW

## Microwave diffuser

The diffuser homogenize the microwave field across all cavity

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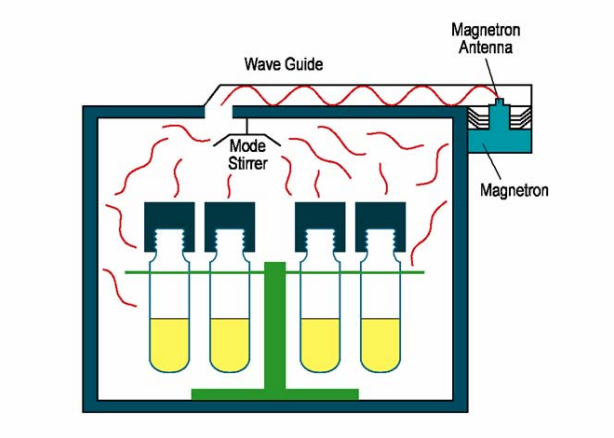
## Microwave a diffuser

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## Pressure responsive door

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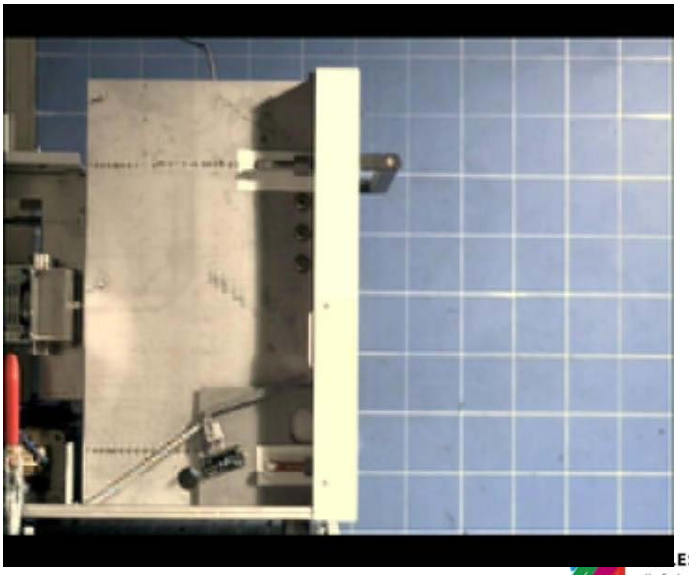


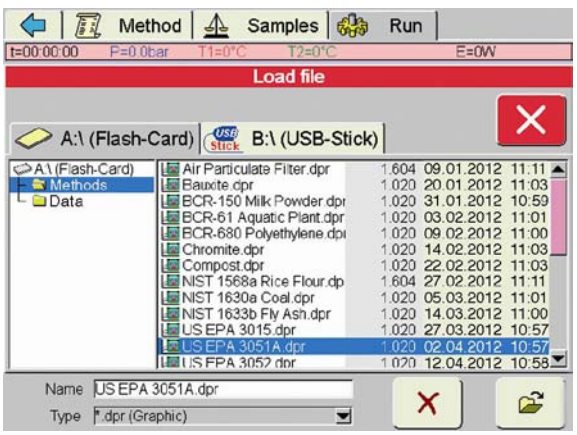
## Door locking system


## Built-in digital camera

# USER INTERFACE

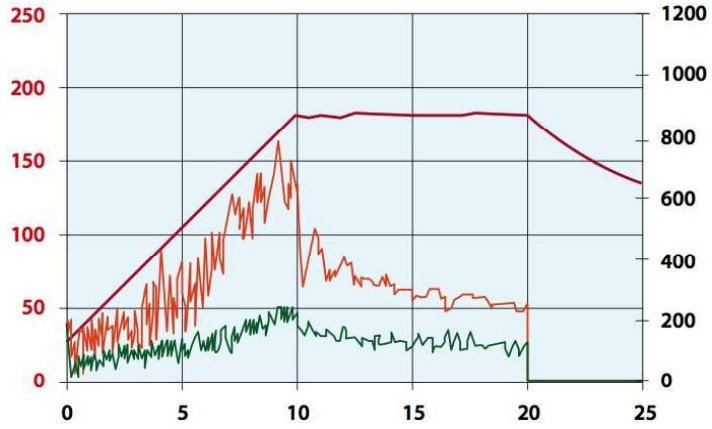
- Touch-screen technology
- Built-in methods library
- One method fits any vessels number
- Better control of exothermal reactions
- Easy data transfer


# Methods Library



# EasyCONTROL software

Same method for any number of vessels

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# EasyCONTROL software

Full control of the exothermal reactions


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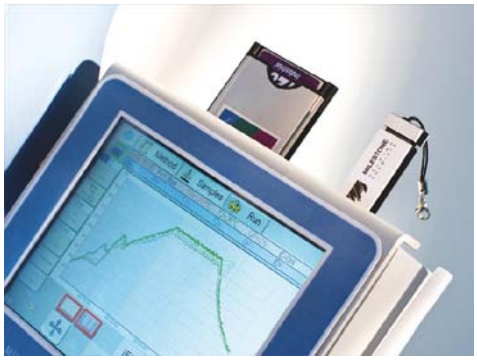
# Easy data transfer



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# REACTION SENSORS

- Direct temperature control
- Contact-less temperature control in all vessels
  - TEMPSURE
- Direct pressure control
- Contact-less pressure control in all vessels

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# Direct temperature control

- Temperature sensor is continuosly controlling the temperature
- Sensor is housed in a PTFE coated ceramic thermowell

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# Temperature control in all vessels (TEMPSURE)

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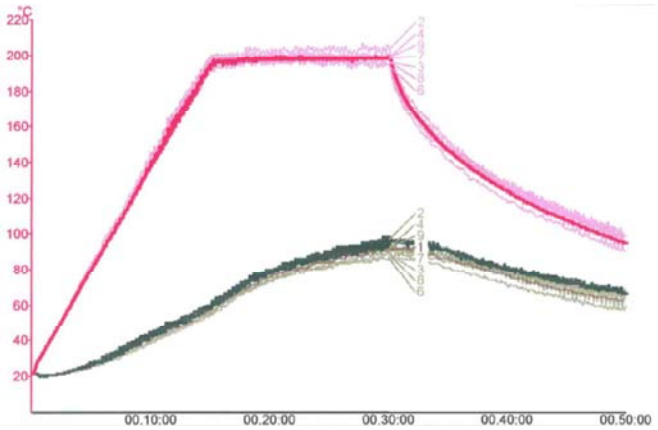
## Temperature control in all vessels (TEMPSURE)

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## Direct pressure control

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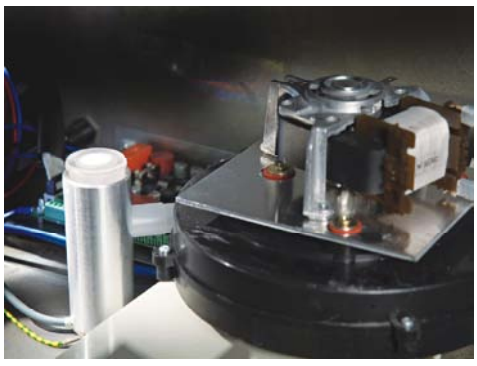
## Pressure control in all vessels (QP)



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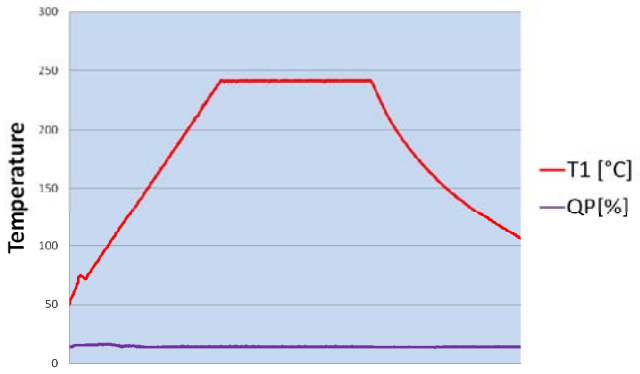
## Pressure control in all vessels (QP)




1 g of infant formula 10 ml of HNO3

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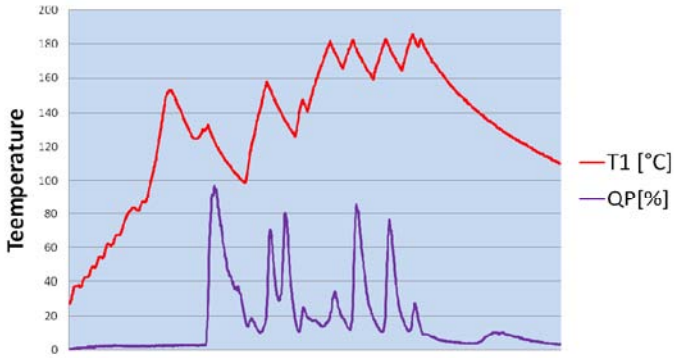
## Pressure control in all vessels (QP)




1,5 g of Infant formula with 10 ml of HNO3

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## VESSEL TECHNOLOGY

- Patented "vent-and-reseal" technology
- Highest temperature and pressure
- Highest safety standards
- Ease of use
- Fast cooling

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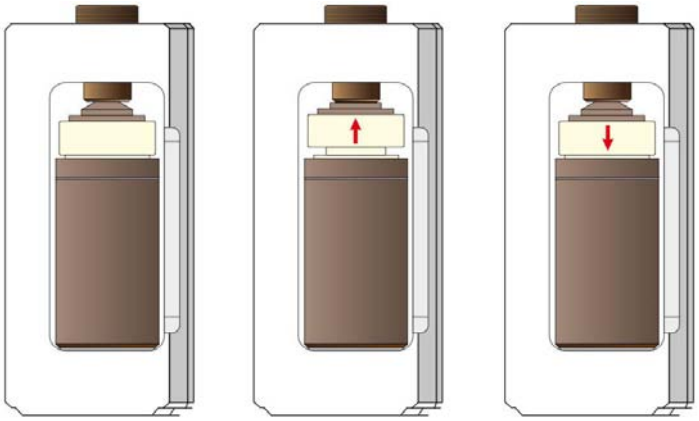
## Vent-and-reseal technology



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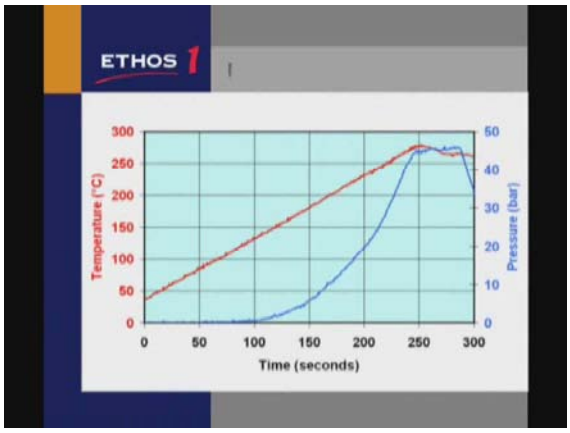
## Vent-and-reseal technology

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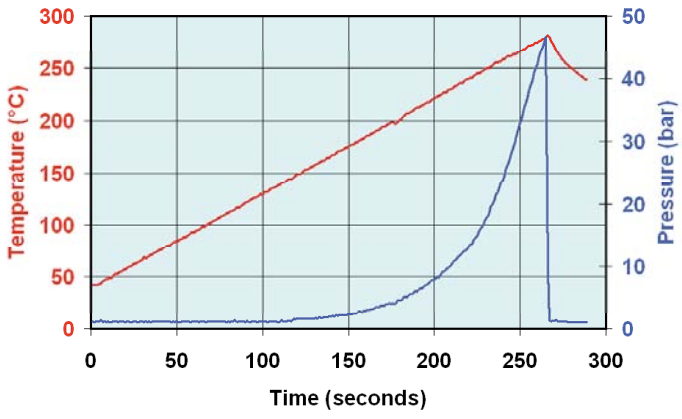
## Burst Disk

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# Microwave FLEXIBILITY

- Close vessel digestion
- Open vessel digestion
- Vacuum evaporation
- Solvent extraction
- Protein Hydrolysis
- Fusion
- Synthesis

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# Microwave Close Digestion

## Acids Chemistry

Non-oxidizing

- Hydrochloric acid
- Hydrofluoric acid
- Phosphoric acid
- Diluted sulfuric acid
- Diluted perchloric acid

Oxidizing

- Nitric acid
- Hot concentrated perchloric acid
- Concentrated sulfuric acid
- Hydrogen peroxide

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# Nitric Acid

- Boiling point is 120°C at 65% concentration
- Poor oxidizing strength at concentrations less than 2 M; oxidizing strength increases with concentration and reaction temperature and pressure
- Most common acid for oxidation of organic matrices

$(CH_2)_X + HNO_3 \rightarrow CO_2(g) + NO_X(g) + H_2O$

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# Nitric Acid

- It dissolves most metals forming soluble nitrates, exceptions are Au and Pt (not oxidated) and Al, B, Cr, Ti and Zr (passivated)
- These metals require acid mixtures or diluted nitric acid
- Often mixed with $H_2O_2$, HCl and $H_2SO_4$
- Available in high purity for trace analysis

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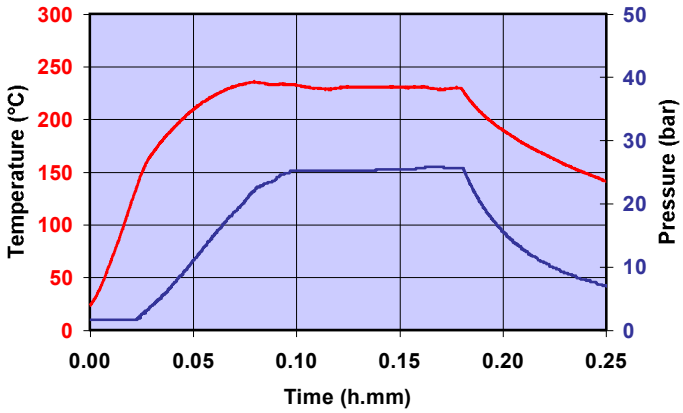
# Nitric Acid

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# Hydrochloric Acid

- Boiling point of azeotropic mixture with $H_2O$ with 20,4% HCl is 110°C
- Available with 38% concentration
- Nonoxidizing
- It dissolves salts of weak acids (carbonates, phosphates) and most metals are soluble with the exception of AgCl, HgCl and TiCl
- Excess of HCl improves the solubility of AgCl, converted into $AgCl_2^-$

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# Hydrochloric Acid

- Strong complexing nature
- Widely used for iron-based alloys because of its ability to hold large amounts of chloro-complex in solution
- Other complexes formed are Ag (I), Au (II), Hg (II), Ga (III), Tl (III), Sn (IV), Fe (II) and Fe (III)
- It does not dissolve oxides of Al, Be, Cr, Ti, Zr, Sn and Sb; sulphates of Ba and Pb, group II fluorides, $SiO_2$, $TiO_2$ and $ZrO_2$

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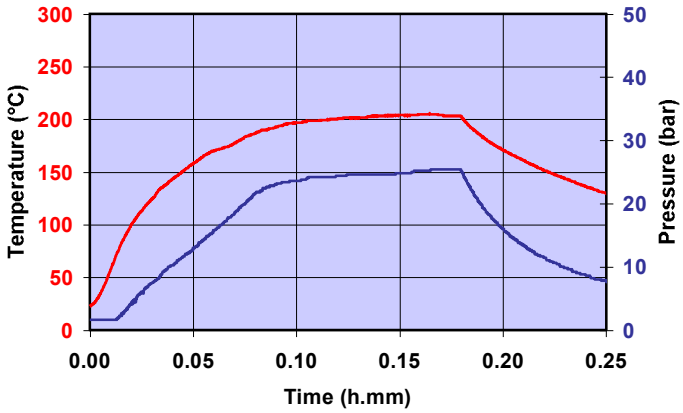
# Hydrochloric Acid

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# Hydrofluoric Acid

Digestion

- Boiling point is 108°C at 40% concentration
- Nonoxidizing, strong complexing nature
- Used in digestion of minerals, ores, soils, rocks and even botanical samples
- Major use is the decomposition of silicates

$$SiO_2 + 6HF \rightarrow H_2SiF_6 + 2H_2O$$

- Often used in combination with $HNO_3$ or $HClO_4$

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# Hydrofluoric Acid

Concentration

- Following dissolution, many analyses require removal of HF to prevent equipment damage or to resolubilize insoluble fluorides

$$H_2SiF_6 \rightarrow SiF_4 + 2HF$$

- Many analytes such as As, B, Se, Sb, Hg, Cr may volatilize

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# Hydrofluoric Acid

Complexation

- Alternative approach to remove HF from the solution, by addition of boric acid
- The following reactions take place

$$H_3BO_3 + 3HF \rightarrow HBF_3(OH) + 2H_2O$$

$$HBF_3(OH) + HF \rightarrow HBF_4 + H_2O$$

- 10-50 times excess boric acid enhances reaction rate

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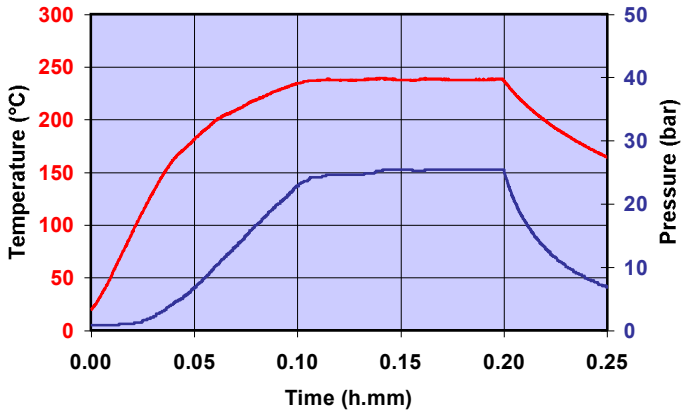
# Hydrofluoric Acid

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# Sulfuric Acid

- Boiling point is 340°C at 98% concentration, exceeding max working temperature of Teflon vessels
- Careful reaction temperature monitoring is required to prevent vessel damages
- It destroys organics by dehydrating action
- Many sulfates are insoluble (Ba, Sr, Pb)

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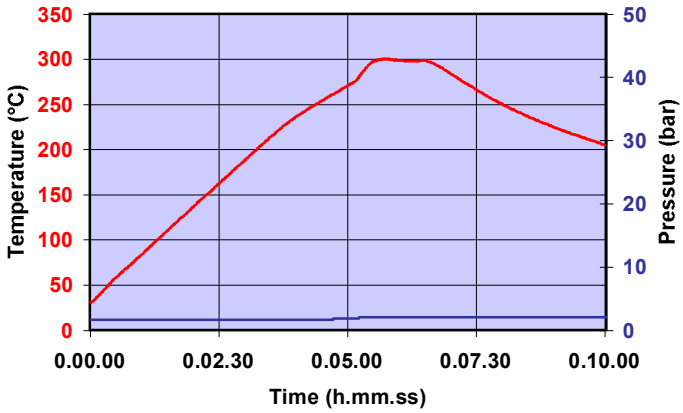
# Sulfuric Acid

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# Perchloric Acid

- Boiling point is 203°C at 72% concentration
- Powerful oxidizing acid when used warm
- Hot and concentrated decomposes violently organic matter
- Nearly all perchlorates are soluble
- $HClO_4$ decomposes at 245°C in microwave closed vessel with dangerous amounts of by-products and tremendous excess pressure

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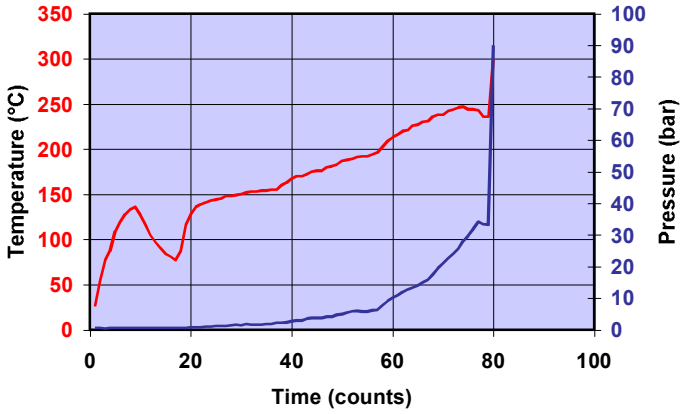
# Perchloric Acid

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# Perchloric Acid

- Rule #1: do not use it
- Use only very diluted perchloric acid
- Mix it with other acids (but never with sulfuric acid)
- Never exceed 200°C
- Use it only to perform a two-step digestion
- Perchloric acid is normally not required for the closed vessel microwave digestion of organic samples

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# Hydrogen Peroxide

- Oxidizing agent

$$2H_2O_2 \rightarrow 2H_2O + O_2$$

- Added to $HNO_3$ it reduces nitrous vapors and it accelerates the digestion of organic samples by raising the temperature
- Typical mixture ratio is $HNO_3$:$H_2O_2$=4:1

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# Organic Samples

- Nitric acid is the most common oxidizing agent used to digest organic samples, according to the following reaction

$$ORG + HNO_3 \rightarrow CO_2 + H_2O + NO_X$$

- Metals are converted into soluble nitrates, available for analysis

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# Temperature

- High fat (cheese, butter, vegetable oil etc.)
  → 180°C
- High protein (bovine, serum, albumin)
  → 160°C
- High carbohydrates (wheat, sugar etc.)
  → 140°C
- Based on sample decomposition with $HNO_3$

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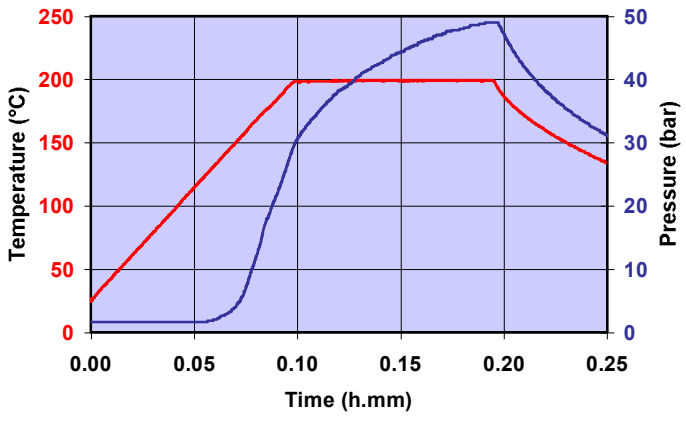
# 0,5 g Olive Oil

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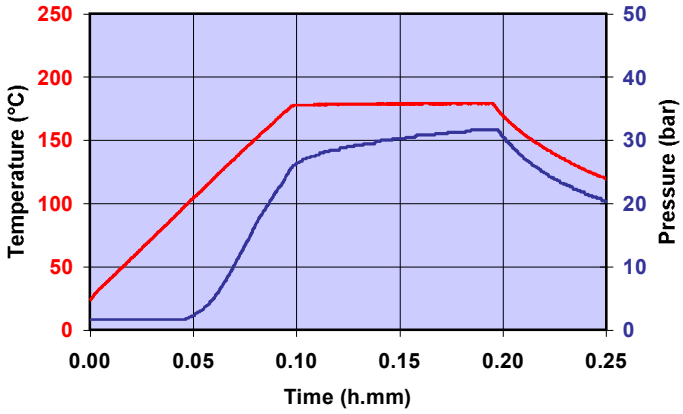
# 0,5 g Milk Powder

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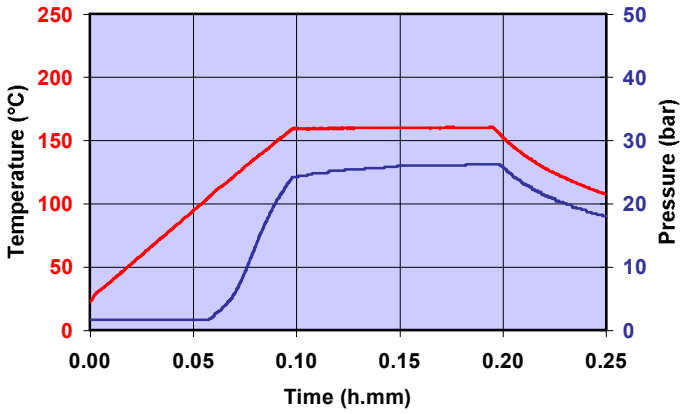
# 0,5 g Noodles

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# Pressure

- Temperature is key
- Pressure is mean
- Microwave heating raises acid temperature and vapor pressure
- Gaseous products ($CO_2$ and $NO_X$) are formed from sample decomposition

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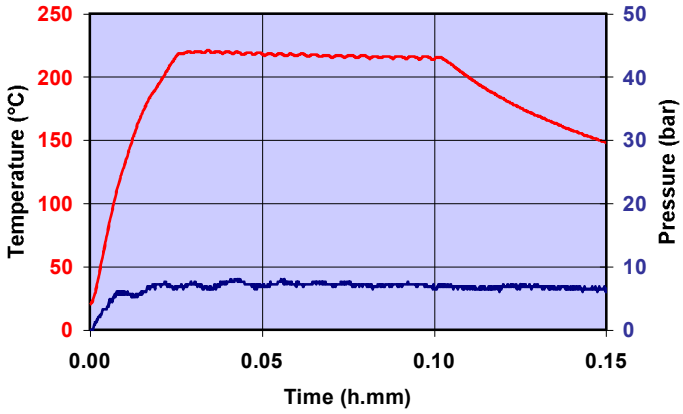
# 0,1 g Milk Powder

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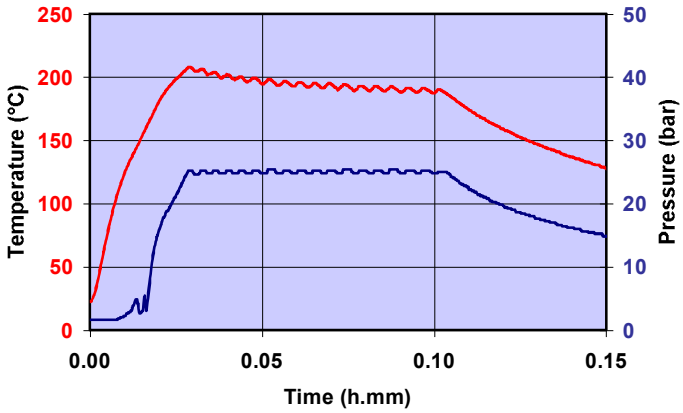
# 0,25 g Milk Powder

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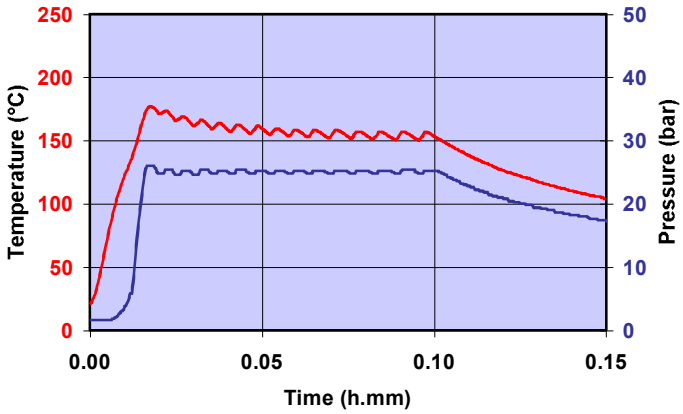
# 0,5 g Milk Powder

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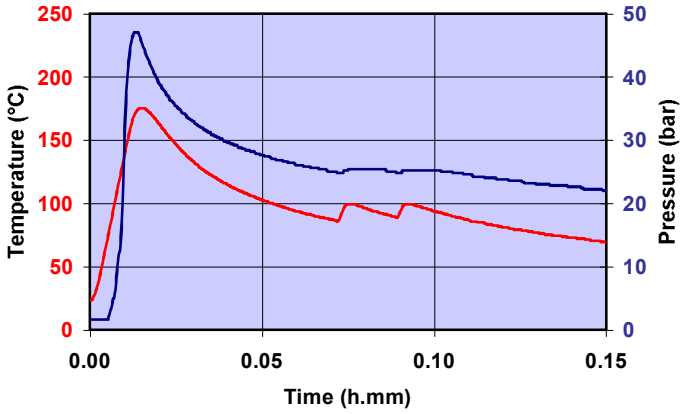
# 1,0 g Milk Powder

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# Milk Powder

| Sample weight | Set temperature | Actual temperature | Set pressure | Actual pressure |
|---|---|---|---|---|
| 0,1 g | 220◦C | 220◦C | 25 bar | ~ 8 bar |
| 0,25 g | 220◦C | ~ 200◦C | 25 bar | 25 bar |
| 0,5 g | 220◦C | ~ 150◦C | 25 bar | 25 bar |
| 1,0 g | 220◦C | ~ 100◦C | 25 bar | Up to 48 bar |

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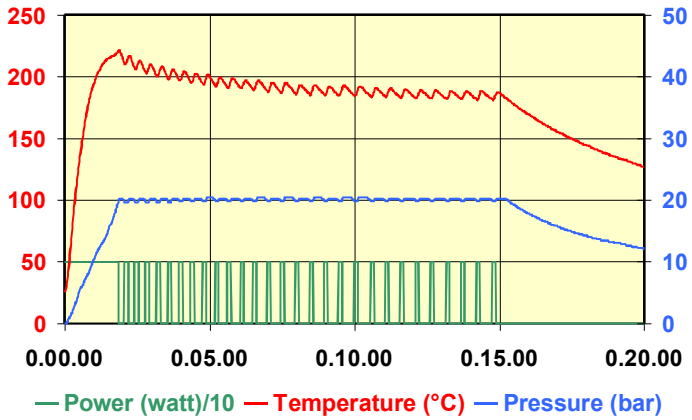
# Limitations of Pressure Control

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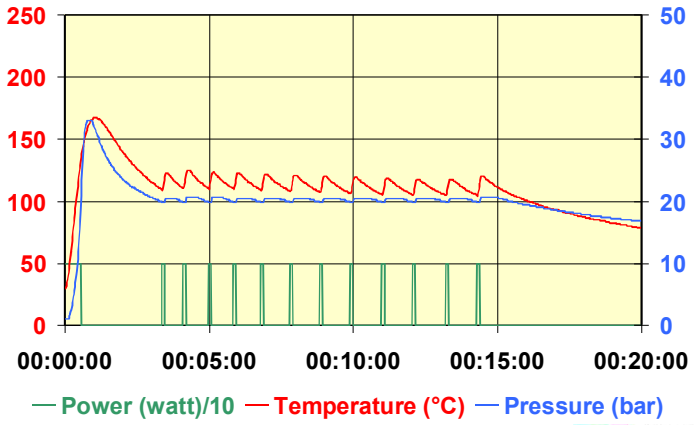
# Limitations of Pressure Control

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# Pressure-based Digestion Quality

- Left
  - 1,0 gram Leaves
    - 20 bar
- Right
  - 0,25 grams Leaves
    - 20 bar

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# Ethos One video

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# What is Next?

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# Current Limitations

- Digestion quality
  - Sample amount
  - Temperature and pressure
- Productivity
  - Sample throughput
  - Disposable vials
- Ease of use
  - Vessels handling
  - Methods library

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# UltraWAVE

The Game Changer in microwave sample preparation


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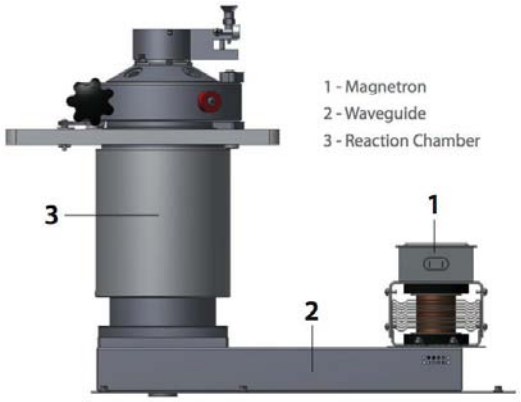
# Single Reaction Chamber




*UltraWAVE Schematic*

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# Single Reaction Chamber

- The microwave cavity is the reaction vessel
- 1500 Watt high microwave power
- Direct microwave coupling
- 990 mL stainless steel reaction chamber
- 900 mL sealed TFM liner inside the chamber

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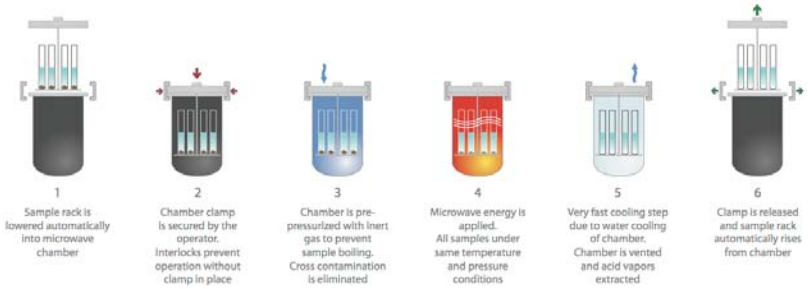
# Operating sequence

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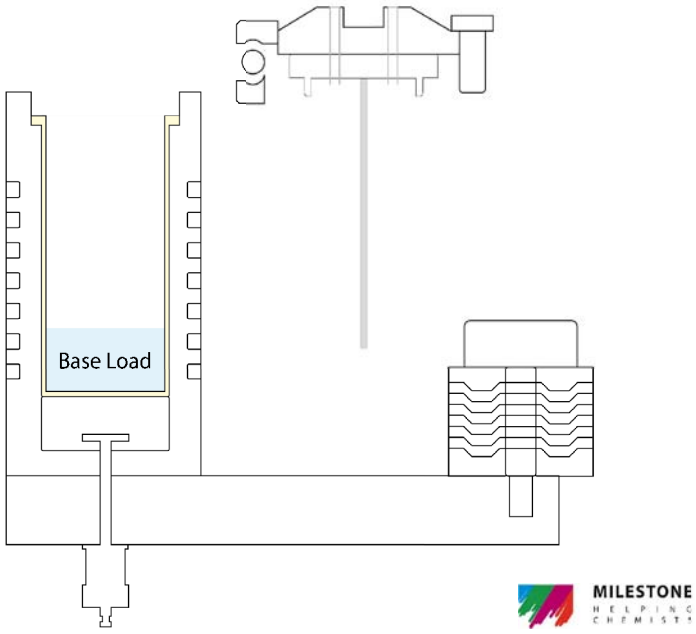
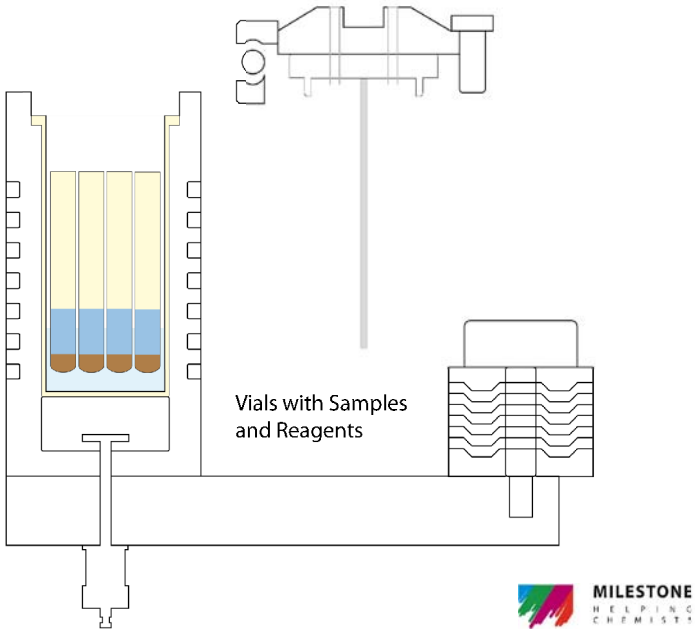
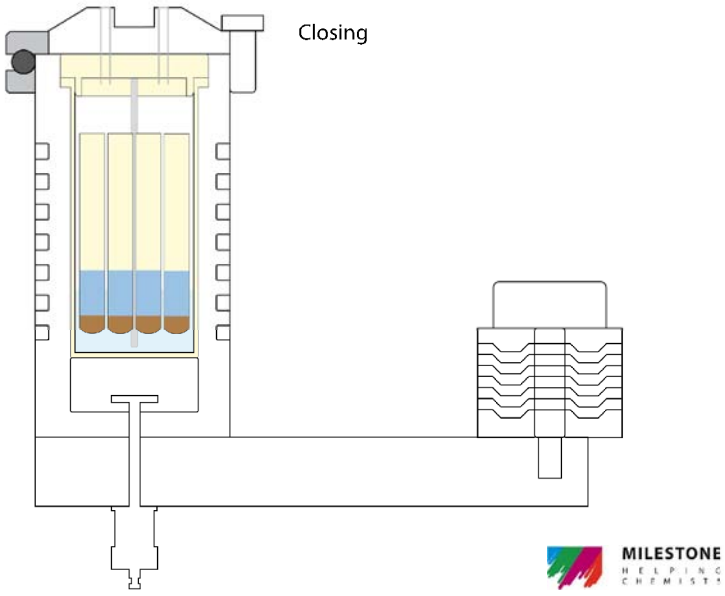
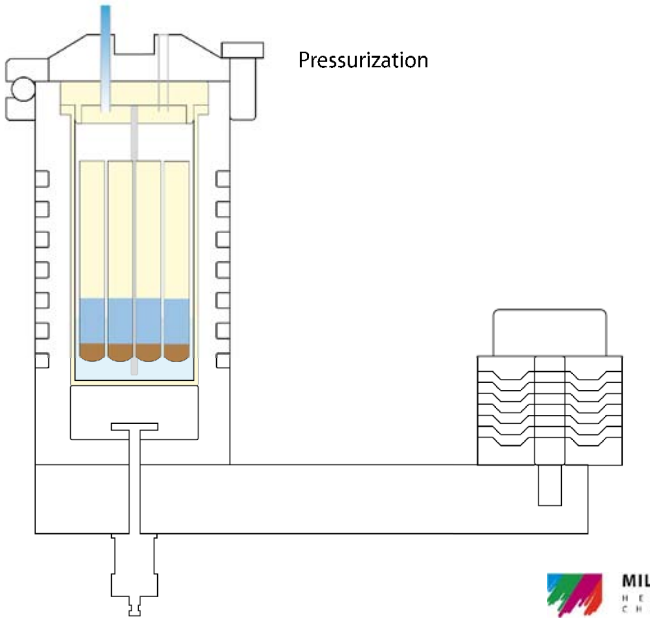

Base Load
MILESTONE


Vials with Samples
and Reagents
MILESTONE


Closing
MILESTONE

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# Pressurization

- Cap for all vials
- Preventing solutions boiling
- Preventing cross contamination

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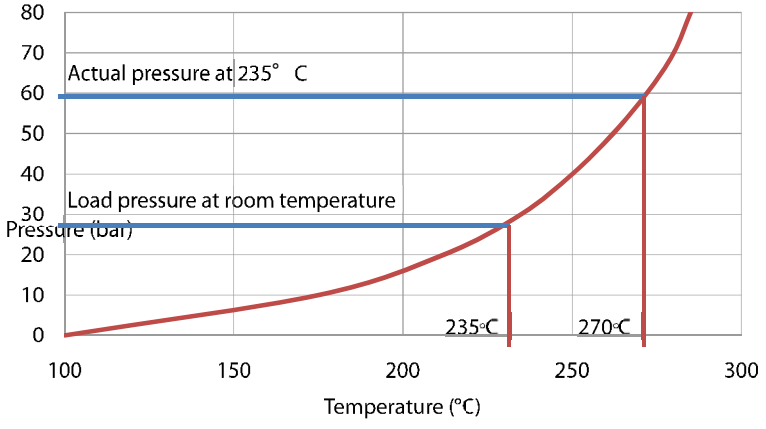
# $H_2O$ P&T

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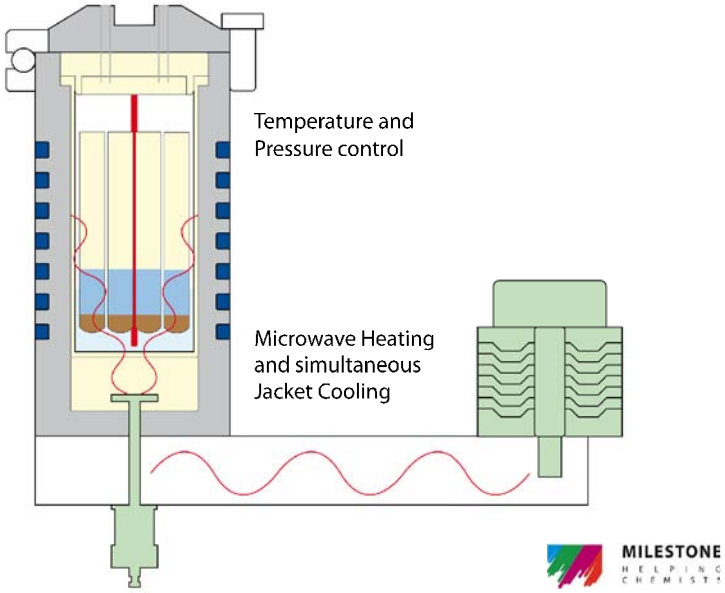


# Reaction Sensors

- Built-in temperature and pressure sensors
- Up to 300C temperature and 200 bar pressure
- No need for a reference vessel
- No need for sensors plug-in
- Any sample combination in the same run
- Same temperature and pressure, regardless of sample type and weight

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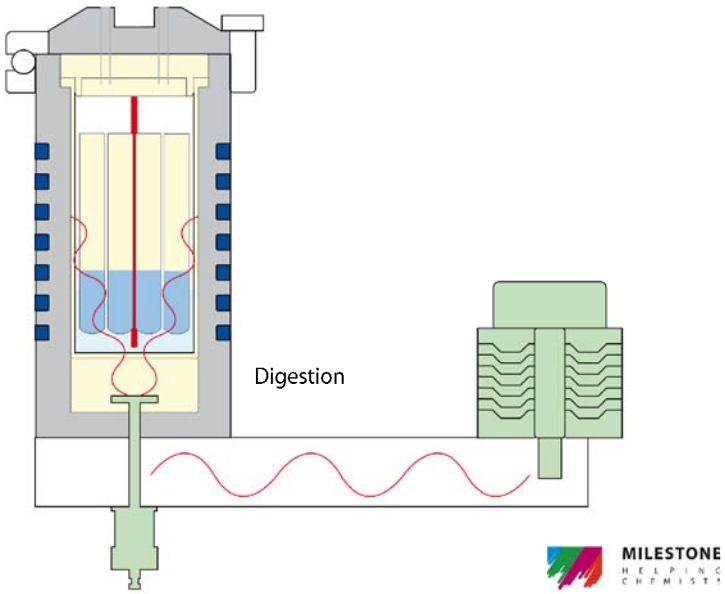
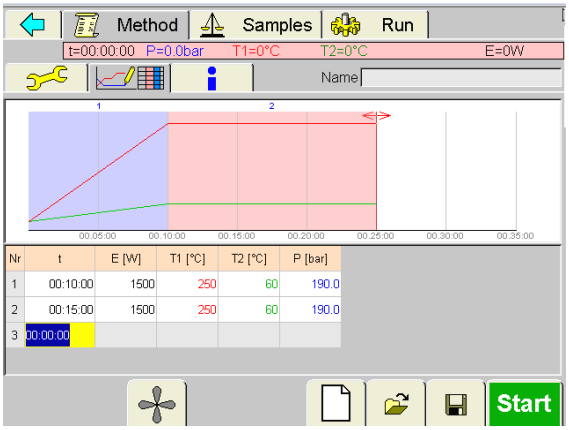



# Method

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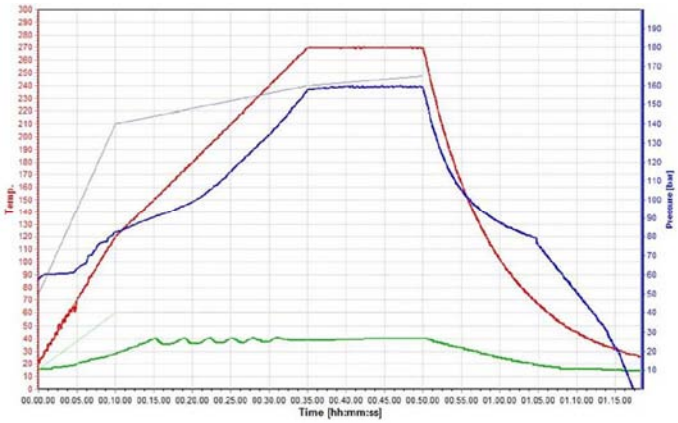
# Run

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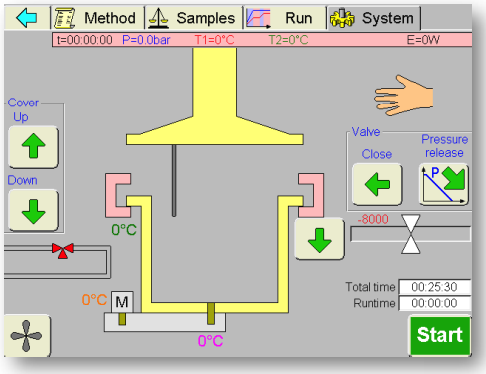
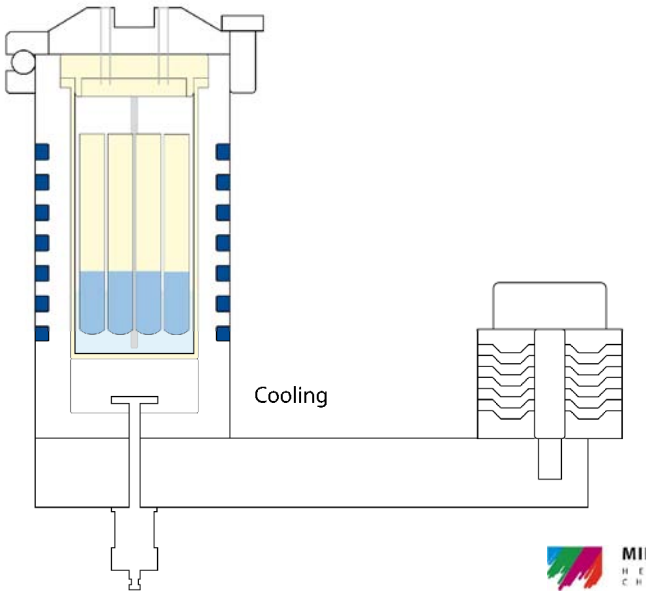
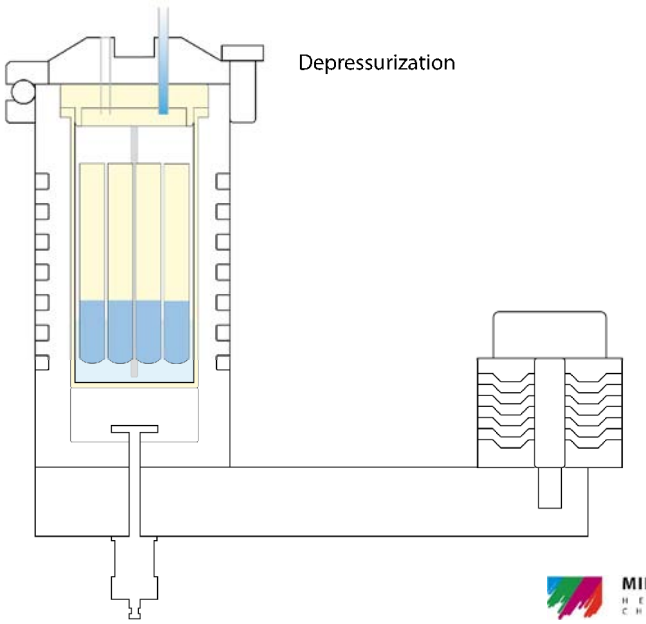
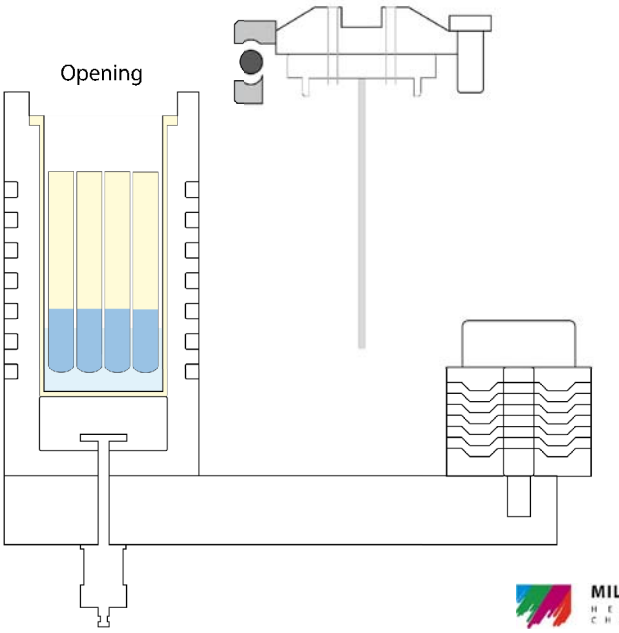
# System


Method
Samples
Run
System
t=00:00:00 P=0.0bar T1=0°C T2=0°C E=0W
Cover
Up
Down
0°C
Valve
Close
Pressure release
-8000
0°C
M
0°C
Total time 00:25:30
Runtime 00:00:00
Start


Cooling


Depressurization





# Expected Benefits

Better digestion quality

Greater ease of use

Enhanced productivity

# Better Digestion Quality

Higher temperature and pressure

Larger sample amount

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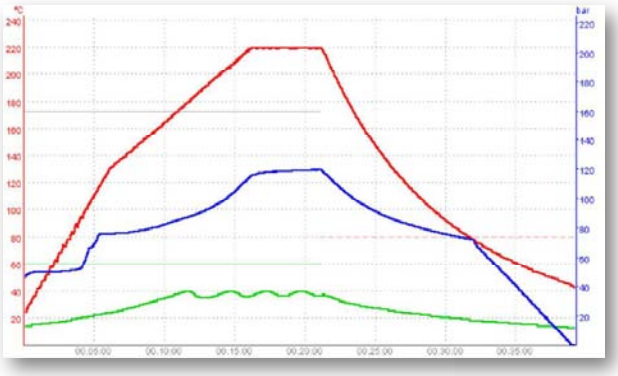
# Larger sample amount

15-position rack with 1,5 g of meat @ 220°C



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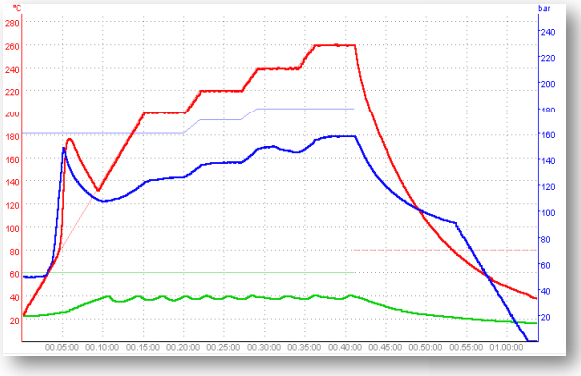
# Larger sample amount

5-position rack with 4g of dry food @ 260°C



Totally 20g of samples!

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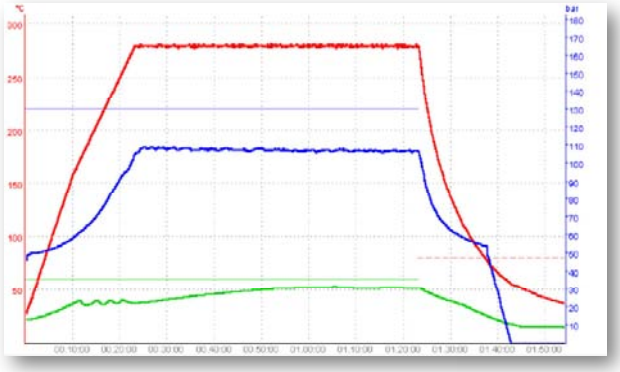
# Digestion of difficult Samples

0,2 g Refractory samples @ 280°C for 1 hour



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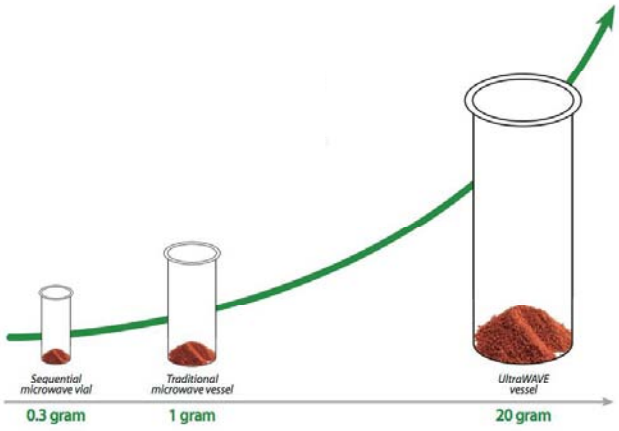
# Larger sample amount

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# Sample amount

- Dry organic material

| Rack | Amount (g) |
|---|---|
| 5 | > 3,0 |
| 15 | ≈ 1,0 |
| 22 | < 0,25 |

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# Recovery Study

**Apple leaves**
*0.5 g sample + 5 ml $HNO_3$*
*Analysis by ICP-MS*

| Element | UltraWAVE | Ring Test |
|---|---|---|
| P | 2,678 | 2,600 |
| K | 21,890 | 20,400 |
| Ca | 8,831 | 8,300 |
| Mg | 2,051 | 1,900 |
| Fe | 92.9 | 94.3 |
| Mn | 79.3 | 75.5 |
| Cu | 9.2 | 10.2 |
| Zn | 45.1 | 43.1 |

*Results in mg/kg*

**Olive leaves**
*0.5 g sample + 5 ml $HNO_3$*
*Analysis by ICP-MS*

| Element | UltraWAVE | ETHOS One |
|---|---|---|
| P | 1,530 | 1,400 |
| K | 12,602 | 13,100 |
| Ca | 21,589 | 23,950 |
| Mg | 1,917 | 2,043 |
| Fe | 88.2 | 88.5 |
| Mn | 28.2 | 26.0 |
| Cu | 31.3 | 33.6 |
| Zn | 18.5 | 17.9 |

*Results in mg/kg*

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Determined concentrations of metals and Se (mean ± standard deviation, $n=3$) in certified reference materials.

| Analyte | Certified values (mg $kg^{-1}$) | | | Determined values (mg $kg^{-1}$) | | |
|---|---|---|---|---|---|---|
| | Apple leaves | Bovine liver | Whole milk powder | Apple leaves | Bovine liver | Whole milk powder |
| Al[f] | 286 ± 9 | 3[a] | 0.9[b] | 268.6 ± 8.7[d] | 2.38 ± 0.19[d] | 1.2 ± 0.1[e] |
| Cu[f] | 5.64 ± 0.24 | 160 ± 8 | 0.46 ± 0.08[c] | 5.8 ± 0.1[d] | 163.5 ± 0.01[d] | 0.56 ± 0.10[e] |
| Fe | 83 ± 5 | 184 ± 15 | 1.8 ± 1.1[c] | 83.5 ± 9.9[d] | 162.1 ± 5.9[d] | ND |
| Mn[g] | 54 ± 3 | 10.5 ± 1.7 | 0.17 ± 0.05[c] | 49.1 ± 3.0[d] | 9.6 ± 0.4[d] | 0.20 ± 0.01[e] |
| Mo[g] | 0.094 ± 0.013 | 3.5 ± 0.3 | 0.29 ± 0.13[c] | 0.080 ± 0.003[e] | 3.6 ± 0.4[e] | 0.33 ± 0.02[e] |
| Rb | 10.2 ± 1.5 | 13.7 ± 1.1 | 16[b] | 12.3 ± 0.1[d] | 17.1 ± 2.0[d] | 18.1 ± 1.1[d] |
| Se | 0.050 ± 0.009 | 0.73 ± 0.06 | 0.131 ± 0.014[c] | ND | 0.75 ± 0.02[e] | ND |
| Sr | 25 ± 2 | 0.136 ± 0.001 | 4.35 ± 0.50[c] | 23.8 ± 2.8[d] | 0.22 ± 0.03[d] | 4.5 ± 0.2[d] |
| Zn | 12.5 ± 0.3 | 127 ± 16 | 28.0 ± 3.1[c] | 10.8 ± 0.1[d] | 97.0 ± 1.8[d] | 25.0 ± 2.1[d] |

ND—not determined.

[a] Noncertified values.
[b] Information concentrations.
[c] Reference concentrations.
[d] Measurement performed by ICP OES.
[e] Measurement performed by ICP-MS.
[f] Y was used as internal standard.
[g] Rh was used as internal standard.

*J.A. Nóbrega et al. / Talanta 98 (2012) 272–276*

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# Ease of Use

Less vessels handling

Mixed samples in the same run

One method for all samples

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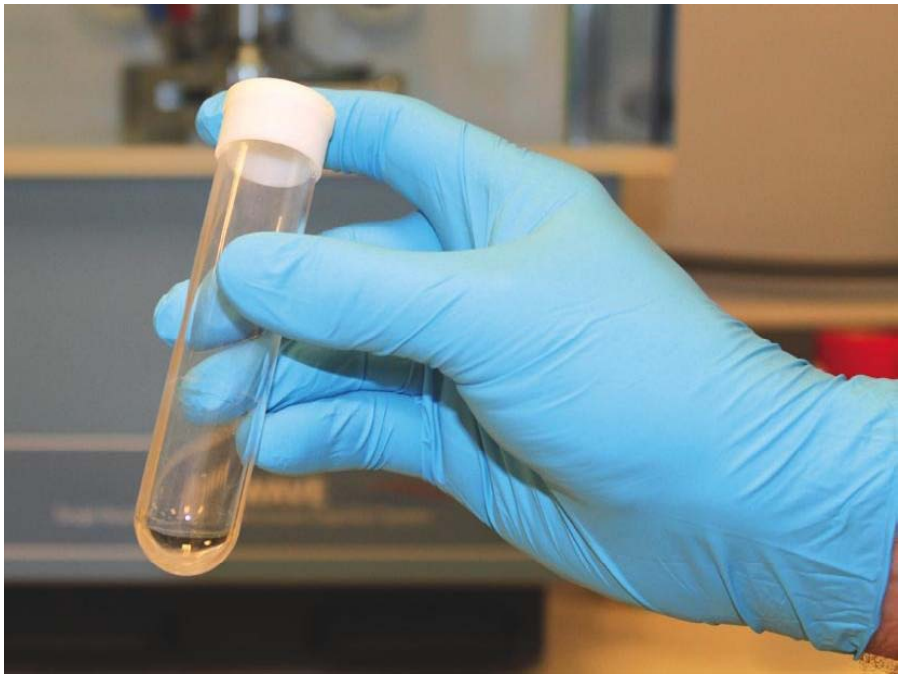
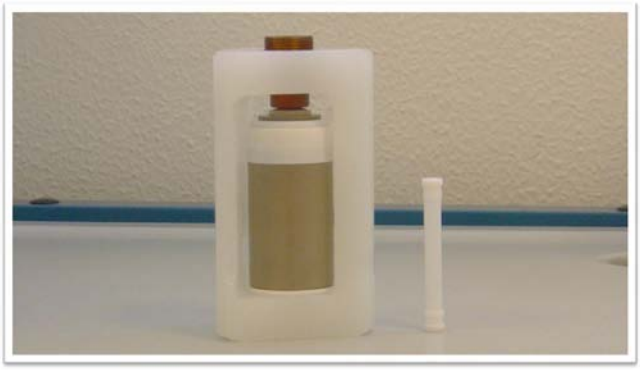


# Vessels vs. Vials



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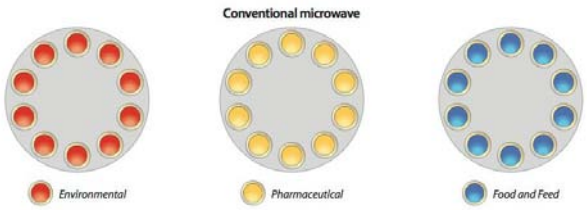
# Conventional microwave

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# One Method for All Samples

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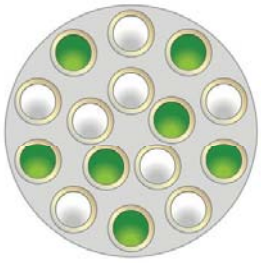
# No Cross Contamination

| Position | Sample | Result (ppb) |
|---|---|---|
| 1 | Blank | 0.02 |
| 3 | Blank | 0.0032 |
| 5 | Blank | 0.001 |
| 7 | Blank | <0.001 |
| 9 | Blank | <0.001 |
| 11 | Blank | <0.001 |
| 13 | Blank | <0.001 |
| 15 | Blank | <0.001 |

*Uncleaned glass vial blanks digested with 110 ppm Hg solutions placed in adjacent vials, showing no evidence of cross contamination*



*Analytical Blank* *110 ppm Hg solution*

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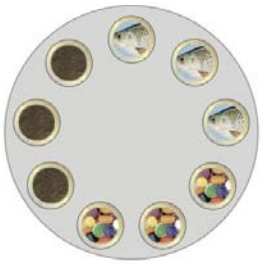
# Complete Recovery

**Certified reference materials**

| Sample | Certified Hg | UltraWAVE Hg |
|---|---|---|
| Fish Protein DORM-3 | 409 ± 27 µg/kg | 393 µg/kg |
| Polyethylene ERM-EC680 | 25.3 ± 1.0 mg/kg | 24.9 mg/kg |
| San Joaquin Soil NIST2709 | 1.4 ± 0.08 mg/kg | 1.4 mg/kg |

*0.5 g sample + 5 ml $HNO_3$*
*Analysis by Milestone DMA-80*



*Fish Protein* *Polyethylene* *San Joaquin Soil*

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# Enhanced Productivity

Fast heating and cooling

High sample throughput

Disposable vials

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# Cooling

- Chamber cooled by closed-loop water cooling system
- UltraWAVE chamber directly connected to a water chiller
- Sensor to continuously monitor and control the temperature of the stainless steel chamber to prevent over-heating

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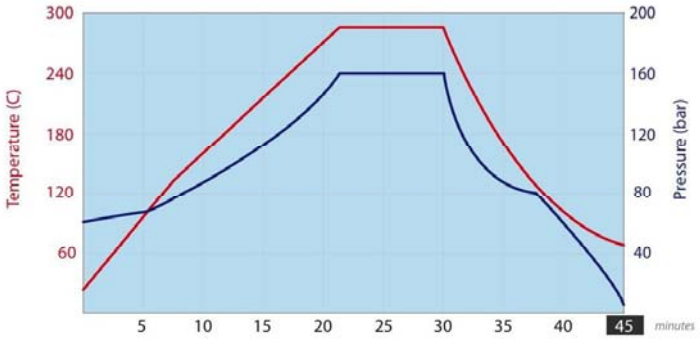
# Fast Heating and Cooling

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# Racks and vials

| Rack Positions | Vials Material | Vials Volume (mL) |
|---|---|---|
| 5 | Glass (disposable)<br>Quartz<br>TFM | Up to 40 |
| 15 | Glass (disposable)<br>Quartz<br>TFM | Up to 15 |

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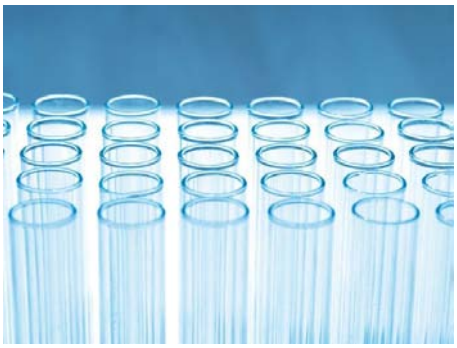
# Disposable glass vials

- Eliminate the cleaning step
- Inexpensive
- Reduce overall sample preparation time



# Blank comparison

| Material | Cleaning before the run | UltraWAVE conditions |
|---|---|---|
| Glass | Not cleaned | 4 ml of HNO3 at 220°C for 20' |
| Quartz | Soacked overnight in acid bath | |
| TFM | | |

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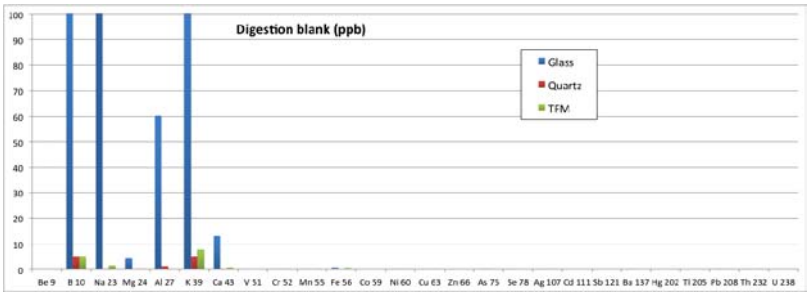
# Blank Comparison Data




Max concentration scale 100 ppb. B, Na, Al, K, Ca observed in glass blank

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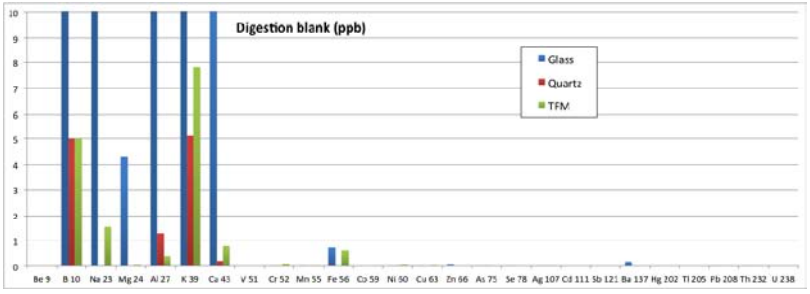
# Blank Comparison Data




Concentration scale zoomed to 10 ppb. B, K at single figure ppb level in quartz and TFM

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# Milestone UltraWAVE

- Better digestion quality
- Greater ease of use
- Enhance productivity

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# UltraWAVE video

## Microwave Open Digestion (MOD)

## Microwave Protien Hydrolysis

# ADVANTAGES of Microwave Protein Hydrolysis

## Time savings

- The total hydrolysis time is much less than the normal analysis time with Milestone instrument, the complete procedure needs less than 45 minutes.
- A first method, which lasts 5 minutes, is used in the sample preparation of "sensitive“ Amino Acids such as Met, Ser, Thr, Tyr, Phe and Arg, which are not thermically degradable.
- a second method of about 25 minutes allows the complete breaking of the aliphatic Amino Acids linkage.

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# ADVANTAGES of Microwave Protein Hydrolysis

## Uniform work conditions

- All samples are processed under equivalent temperature conditions assuring a noticeable reproducibility in analytical data

## Inert/Anaerobic environment

- The hydrolysis is performed under inert, anaerobic conditions in order to avoid oxidative degradation of amino acids.
- The Milestone system, thanks to the special VS-5 valve, offers the possibility to work under vacuum and with nitrogen, a complete answer to these needs.

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# ADVANTAGES of Microwave Protein Hydrolysis

## No contamination

- The hydrolysis is carried out in quartz vials that can be directly used by the HPLC auto-sampler, thereby eliminating any possible contamination or analytical loss.

## Control of Hydrolysis conditions

- The continuous monitoring of the temperature allows the operation in controlled and repeatable conditions.

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# ADVANTAGES of Microwave Protein Hydrolysis

## Full safety

- The patented MDR technology has been in used for several years for applications much more dangerous than hydrolysis with diluted hydrochloric acid at 160°C.
- The system is guaranteed to be fully within the most common safety norms.

## Easy operation

- All sample vials are loaded in a single rotor.

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# ADVANTAGES of Microwave Protein Hydrolysis

## Complete Documentation

- The softWAVE software completely documents every hydrolysis parameter.

LESS THAN 45 MINUTES INSTEAD OF 24 TO 72 HOURS!

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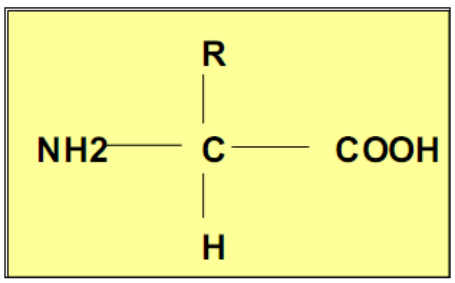
## Structure and Nomenclature of amino acids

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## Structure and Nomenclature of amino acids

| Name | Symbol (3 letter) | Symbol (1 letter) | Molecular Weight | Side chain (R group) |
|---|---|---|---|---|
| Aspartic Acid | Asp | D | 133 | CH2-COOH |
| Glutamic Acid | Glu | E | 147 | CH2-CH2-COOH |
| Alanine | Ala | A | 89 | CH3 |
| Asparagine | Asn | N | 132 | CH2-CONH2 |
| Cysteine | Cys | C | 121 | CH2-SH |
| Cystine | CySS | | 240 | CH2-S-S-CH2-CH(NH2)-COOH |
| Glutamine | Gln | Q | 146 | CH2-CH2-CONH2 |
| Glycine | Gly | G | 75 | H |
| Isoleucine | Ile | I | 131 | CH(CH3)-CH2-CH3 |
| Leucine | Leu | L | 131 | CH2-CH(CH3)(CH3) |
| Methionine | Met | M | 149 | CH2-CH2-S-CH3 |
| Phenylalanine | Phe | F | 165 | CH2-C6H6 |
| Serine | Ser | S | 105 | CH2OH |
| Threonine | Thr | T | 119 | CHOH-CH3 |
| Tryptophan | Trp | W | 204 | CH2-C8H5N |
| Tyrosine | Tyr | Y | 181 | CH2-C6H5OH |
| Valine | Val | V | 117 | CH(CH3)(CH3) |
| Arginine | Arg | R | 174 | CH2-CH2-CH2-NH-C(NH)-NH2 |
| Histidine | His | H | 154 | CH2-C3H2N2 |
| Lysine | Lys | K | 146 | CH2-CH2-CH2-CH2-NH2 |
| Ornithine | Orn | O | 132 | CH2-CH2-CH2-NH2 |

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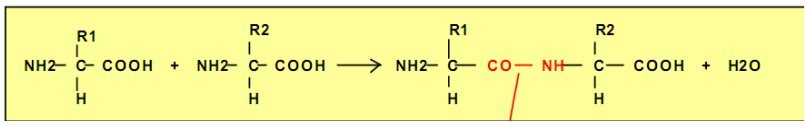
# Protein Hydrolysis




Peptide Linkages

A protein is a great polypeptide, with more than 20 amino acids.

The analysis of the amino acid composition is carried out after the hydrolysis of the peptide linkages.

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# Protein Hydrolysis

- The hydrolysis with HCl is the most frequently used hydrolysis technique, both under reflux or at 110°C in sealed quartz tubes in which air is previously evacuated
- The hydrolysis time varies from 18 to 72 hours, depending on the type of peptides linkage.
- Hydrolysis under different conditions (and with differentiated reactive added) must be practised to obtain a complete screening of the amino acids present

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# Principle of operations

- Weight the samples directly in the 4ml quartz vials
- Wetted with a few drops of HCl 6N.
- The 330ml PTFE vessel is partially filled with about 30ml of HCl 6N.
- fitted in the PTFE vessel which is inserted in the safety shield.
- cover and complete with the temperature sensor, is placed on the vessel.
- closed under pressure by means of the torque wrench

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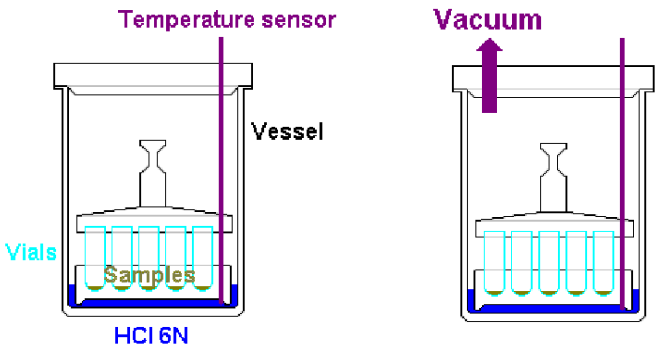
# Principle of operations

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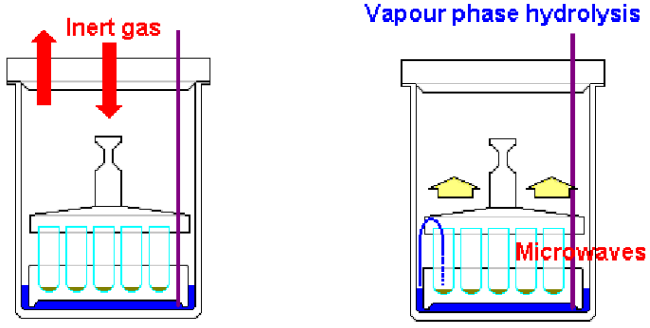
# Principle of operations

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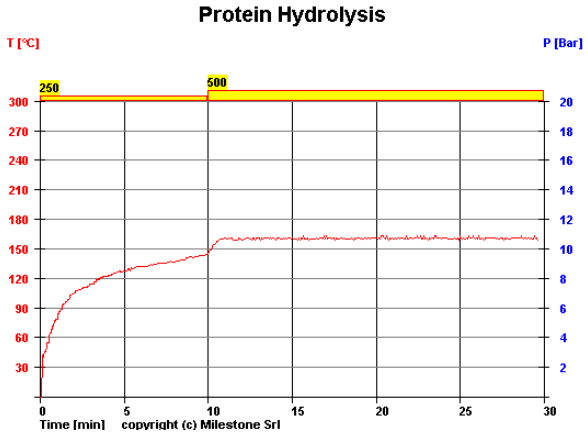
# Principle of operations

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# Applications

*Acid protein hydrolysis in vapour phase with Milestone instrument*

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# Applications

* Sample preparation
  - weigh the solid sample (0.15-1.5 mg) in 1.5 ml test tube
  - wet the sample with 40 ul of HCl 6N
  - place the samples in Teflon rotor containing 30 ml of HCl 6N
  - insert in microwave oven
  - make vacuum and let in Nitrogen

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# Applications

* Program temperature and power of microwave oven
  - 10 minutes at 250W 160◦C
  - 30 minutes at 500W 160◦C
  - 15 minutes ventilation
* Hydrolization treatment
  - filter the samples diluted with water
  - fill up to a final volume of 500 ul

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# Result

| | Casein | | Glue | | Albumen | | Yolk | |
|---|---|---|---|---|---|---|---|---|
| *Ratios* | *average* | *sd* | *average* | *sd* | *average* | *sd* | *average* | *sd* |
| Glu/Asp | 3,1 | 0,18 | 1,8 | 0,12 | 1,1 | 0,1 | 1,2 | 0,12 |
| Leu/Ala | 3 | 0,31 | 0,4 | 0,02 | 1,5 | 0,13 | 1,6 | 0,1 |
| Val/Ala | 2,2 | 0,22 | 0,3 | 0,02 | 1,2 | 0,11 | 1,1 | 0,11 |
| Ala/Phe | 0,6 | 0,07 | 3,9 | 0,25 | 0,9 | 0,14 | 1,2 | 0,25 |
| Leu/Ile | 1,7 | 0,04 | 2,3 | 0,04 | 1,6 | 0,05 | 1,6 | 0,26 |
| Gly/Ile | 0,4 | 0,08 | 15,9 | 1,59 | 0,6 | 0,09 | 0,6 | 0,09 |
| Ala/Gly | 1,4 | 0,2 | 0,4 | 0,02 | 1,8 | 0,11 | 1,8 | 0,11 |
| Ser/Ala | 1,9 | 0,26 | 0,3 | 0,05 | 0,9 | 0,13 | 1,7 | 0,27 |
| Ser/Ile | 1,1 | 0,23 | 1,7 | 0,34 | 1 | 0,14 | 1,6 | 0,31 |

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# Microwave Vacuum Evaporation

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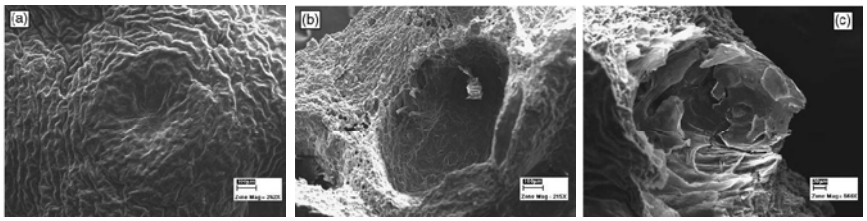
# Microwave Solvent Extraction

## SFME Technology




**Untreated** | **MW (30 min)** | **HD (3 h)**

Ferhat M.A., Meklati B.Y., Smadja J., Chemat F., *Journal of Chromatography* A, (2006) 1112: 121

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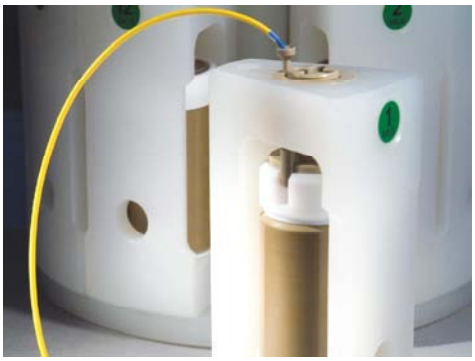
# Microwave Solvent Extraction



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# Microwave Fusion


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# Milestone Product Line

Digestion
Clean Chemistry
Extraction
Ashing
Synthesis
Mercury

Milestone's waves of innovations

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## Digestion

ETHOS One

START D

UltraWAVE

UltraCLAVE

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## Clean Chemistry

TraceCLEAN

DuoPUR

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# Extraction

START E

NEOS

NEOS GR

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# Ashing

PYRO 260

PYRO XL

PYRO SA

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# Synthesis

StartSYNTH

MicroSYNTH

RotoSYNTH

SynthWAVE

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# Mercury

DMA-80

DMA-80L

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Great products deserve
great stage

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## New Milestone Lab Showroom

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# To be recognized on the market *
# you need unique products

* and to have a decent gross margin

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## Milestone has unique products!

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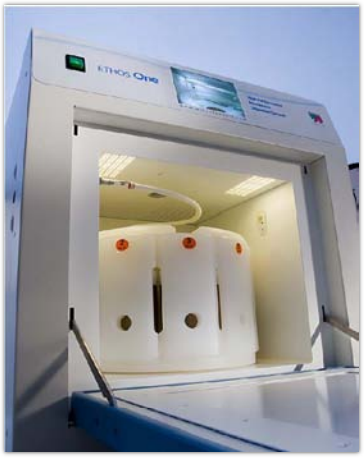
## The NEW ETHOS One

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# TWISTER

## Vessel Handling Module

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# NEOS

- **NEOS**
  Solvent-Free Microwave Extraction (SFME) of Essential Oil

- **NEOS-GR**
  Rapid, Solvent-Free Extraction by Microwave Hydro-diffusion and Gravity (MHG)

# NEOS GR

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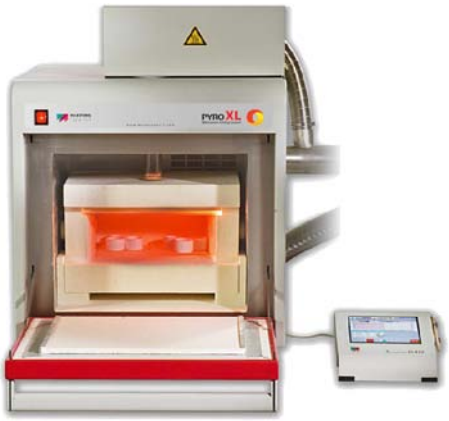
# PYRO XL

## Microwave Ashing System for Extra Large Sample Amounts



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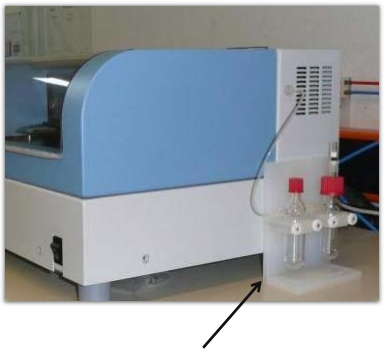
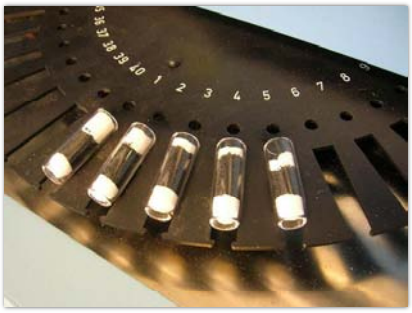
# DMA-80 GAS ACCESSORIES

## GAS KIT



## SORBENT TRAPS



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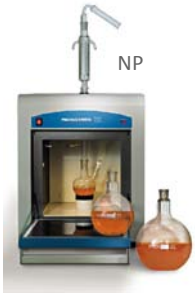
# NEW SYNTHESIS KIT




Pack 1


Pack 2


Pack 3


Pack 4

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