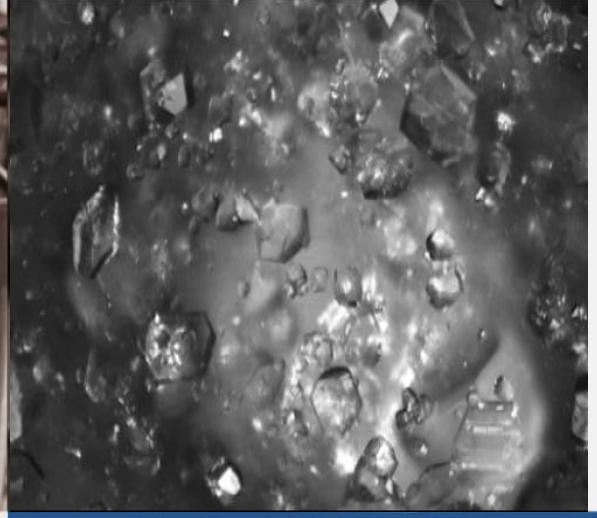
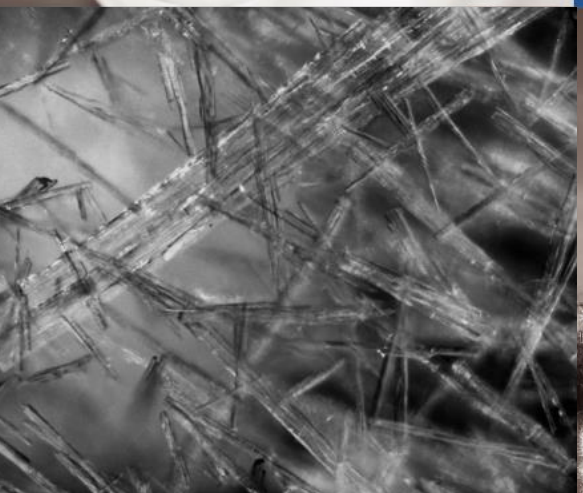




Modern microwave digestion techniques

Tomi Kemppinen




MILESTONE
HELPING CHEMISTS

WHAT CAUSE MOST ERRORS IN CHEMICAL ANALYSIS?

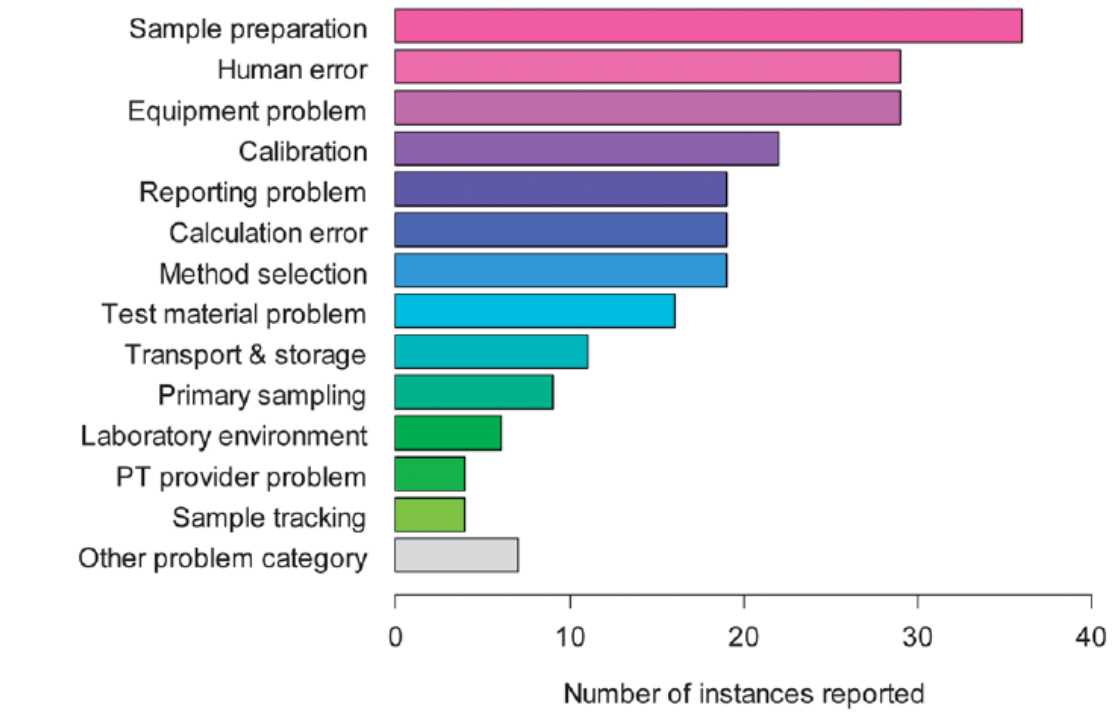


Fig. 1 Causes of error in chemical analysis.

Anal. Methods, 2013, 5, 2914

TRACE METALS ANALYSIS INSTRUMENTATION

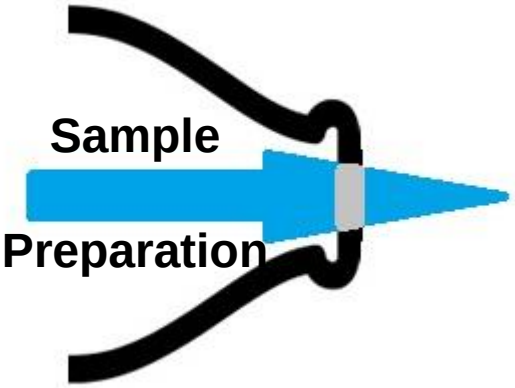
Trace metals analysis can be performed by:

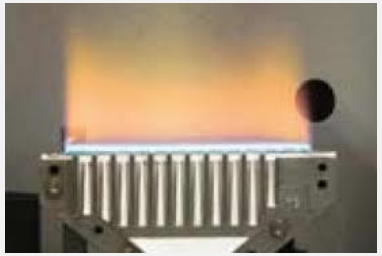
- AAS; GF-AA
- ICP; ICP-MS

There is a significant transition to ICP-MS (trace analysis) due to:

- New regulation (e.g. USP)
- Demand of lower detection limits
- Higher productivity (than GFAA)

THE BOTTLENECK





- Modern analytical equipment gives an accurate analysis in few minutes/seconds. The conventional sample preparation requires: time, reagent consumptions and handling
- Analytical measurement is as good as the sample preparation

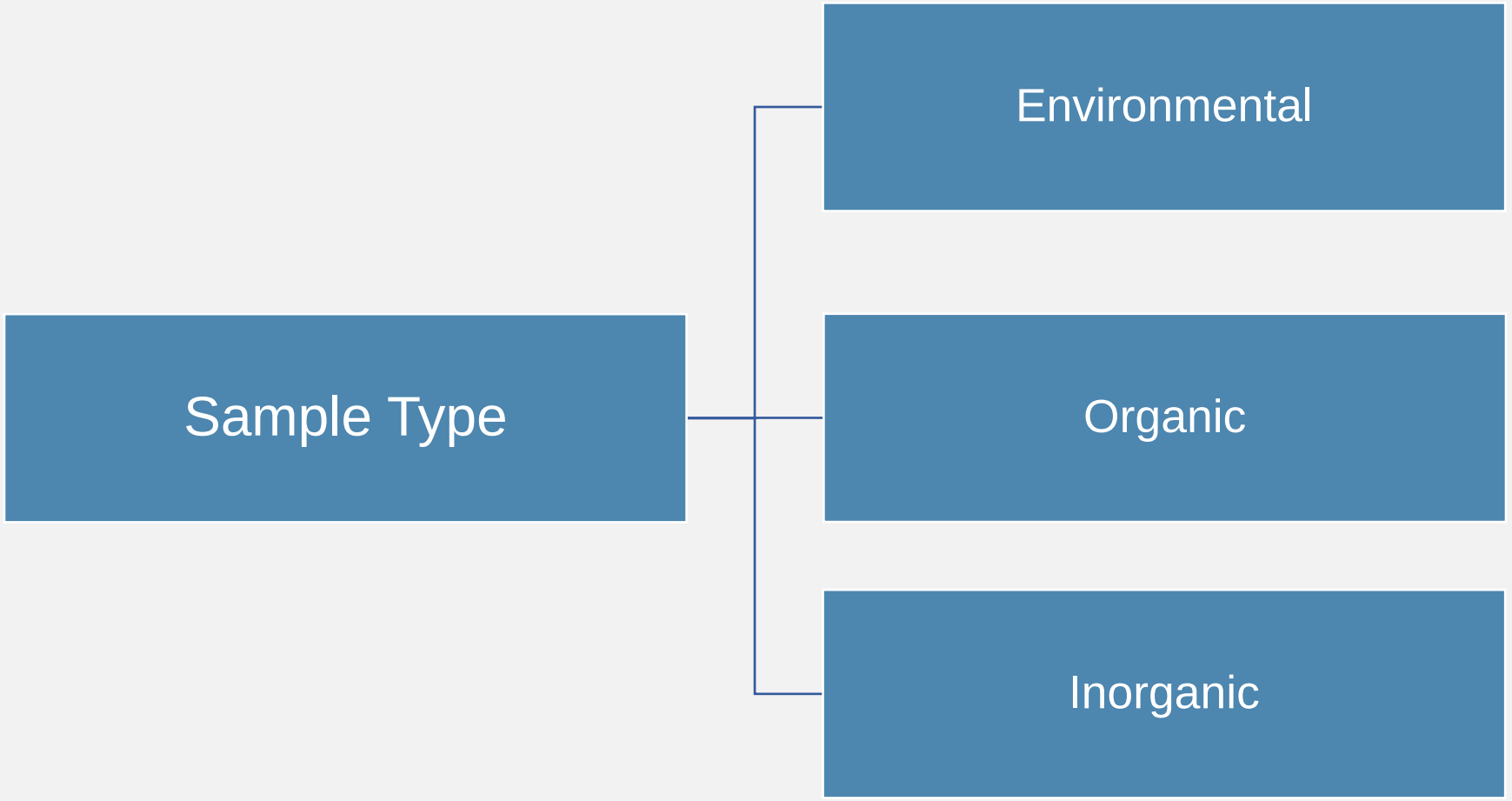
WHY MICROWAVE CLOSED-VESSEL DIGESTION

- Speed/ productivity
  - Microwave closed vessel digestion allow fast digestion of multiple samples simultaneously
- Digestion quality
  - Microwave closed-vessel allow to work at high T&P enhancing the digestion quality
- Safety
  - Microwave hardware ensure high level of safety
- Ease of use
  - Built-in application libraries, easy handling
- Full control
  - Temperature and Pressure control

THE KEY PARAMETERS IN SAMPLE PREPARATION

- Sample type
- Sample weight
- Acid chemistry
- Digestion conditions (temperature and pressure)

SAMPLE TYPE

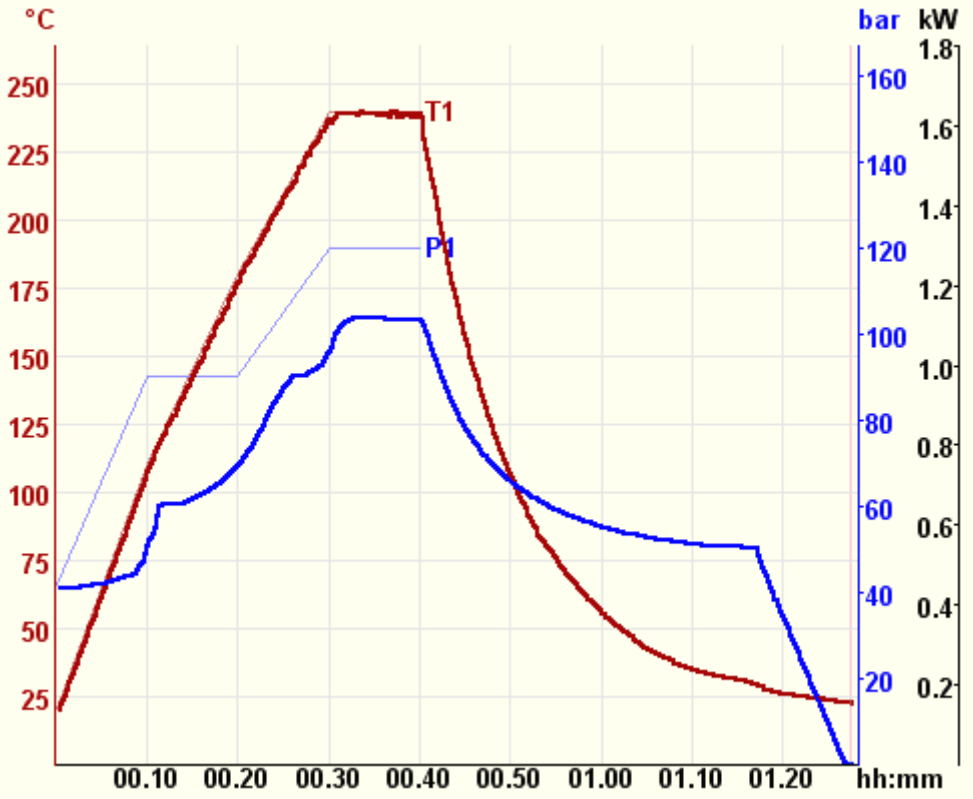


SAMPLE AMOUNT vs PRESSURE

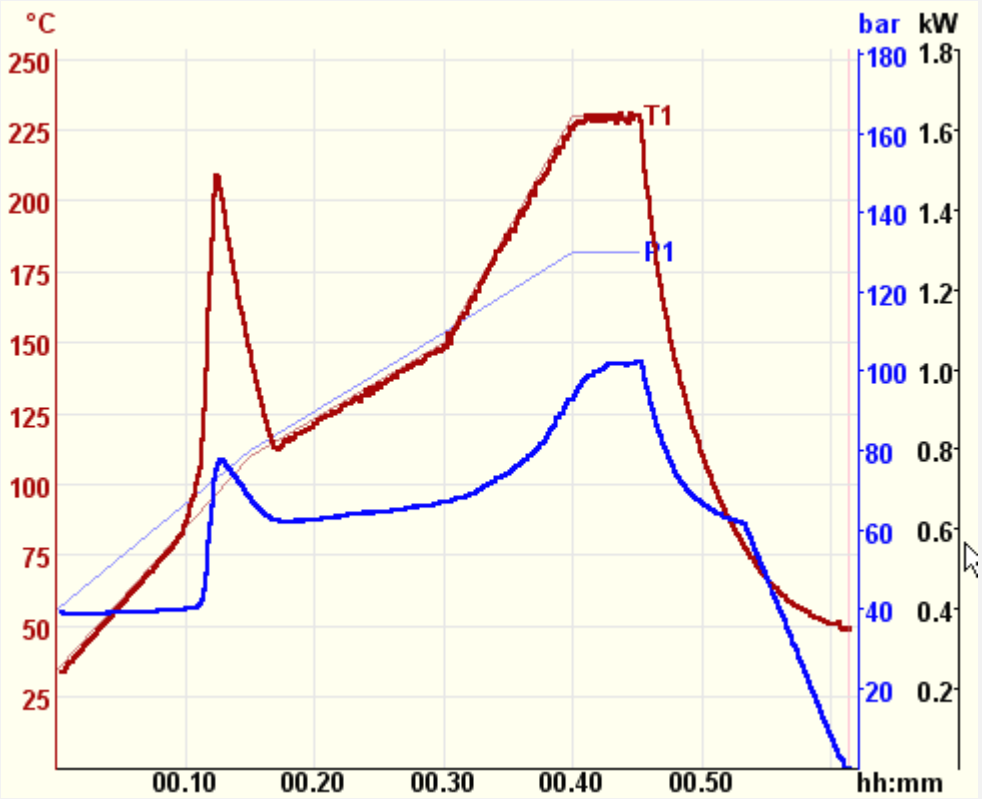
- The sample amount directly impact the maximum pressure
- Vessels and rotors must be able to handle the pressure generated
- Releasing pressure during the run could lead to:
  - Loss of volatile elements
  - High amount of acid fumes in the cavity
  - Releasing pressure is not in compliance with official methods

ORGANIC SAMPLE REACTIVITY

Medium reactivity
(low mass of food samples < 0,5 g)



High reactivity
(high mass of food samples > 4 g)



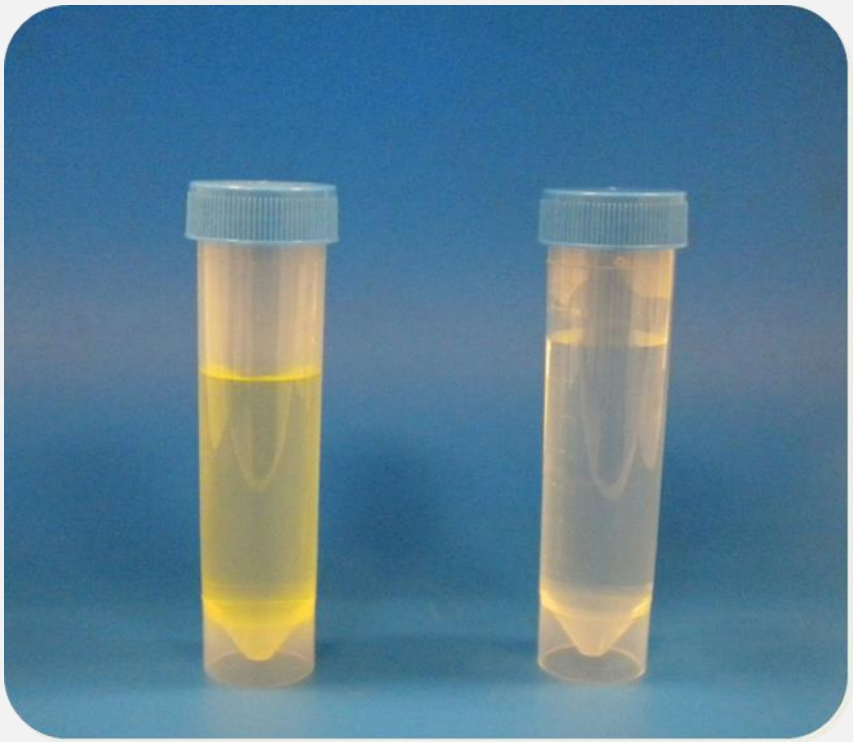
THE EFFECT OF THE TEMPERATURE IN A DIGESTION PROCESS

Test conditions	Test 1	Test 2
Sample	Baby food	Baby food
Sample amount	0,5g	0,5g
Acid mixture	10mL of HNO3 65%	10mL of HNO3 65%
Vessel Volume	100mL	100mL
Digestion time	20 minutes	20 minutes
Set Temperature	**180°C** 32 bar	**200°C** 45 bar

THE EFFECT OF THE TEMPERATURE IN A DIGESTION PROCESS

1. Incomplete digestion due to low T and P (180°C and 32 bar)

2. Complete digestion thanks to the high T and P (200°C and 45 bar)



DIGESTION GUIDELINES

	Typical samples	Typical acid mixture	Typical working T	Comments
Environmental	Soils, sediments, waste water	HNO_3 or HNO_3 and HCl	175°C-180°C	Generates low pressure
Food, beverage and feed	Fruits, meat, juices, cereals, butter	HNO_3, H_2O_2, HCl	180°C-200°C	Often reactive samples, generate high P, HCl used for some elements
Specialty chemicals	Polymers, additives	HNO_3, H_2O_2, HCl	200°C-250°C	Challenging samples, reactive and often requires high T to achieve complete digestion
Pharmaceutical	API's, drugs	HNO_3, H_2O_2, HCl	180°C-230°C	Often reactive samples, generate high P, HCl used for some elements
Petrochemical/ Energy	Coal, coke, crude oil, heavy oil	HNO_3, H_2O_2, HCl	200°C-250°C	Challenging samples, reactive and often requires high T to achieve complete digestion
Geological	Ores, rocks	HNO_3, HCl, H_3PO_4, HF	200°C-260°C	Low P, complex sample composition requires various acids
Metals, alloys	Precious metals, catalysts	Aqua regia, HNO_3, HCl, HF	200°C-280°C	Often requires high T for long time

TARGET OF SAMPLE PREPARATION

1. Achieve complete digestion and recovery of the target elements based on your analytical equipment (application & performance)
2. Ensure to match your productivity needs (throughput)
3. Easy implementation of the system into your lab workflow (ease of use)
4. Ensure low running cost (ROI & reliability)
5. Get premium support (Expertise and know-how)

ETHOS UP

High Performance Microwave Digestion System

TEMPERATURE CONTROL

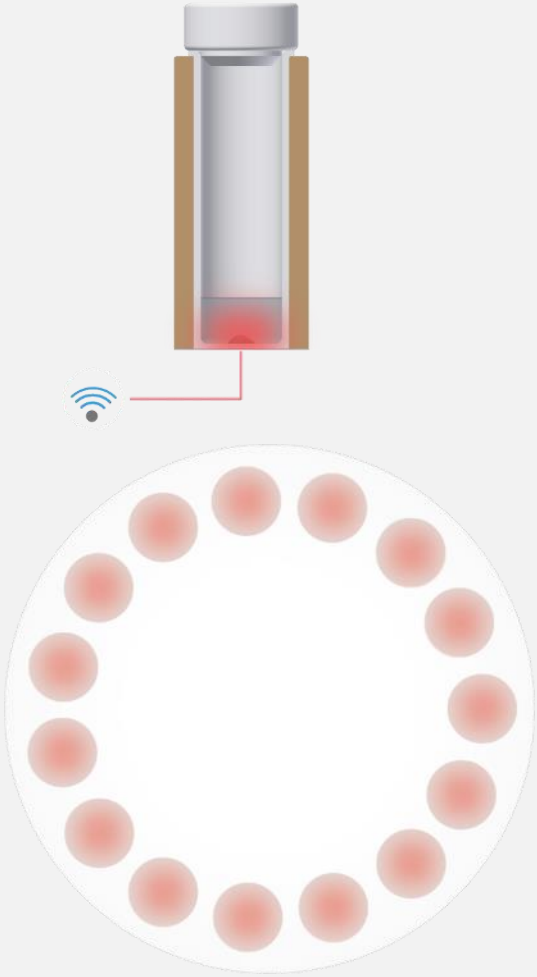
- Temperature is the most important parameter in a digestion process
- Temperature affects the digestion quality
- The temperature control enables to simplify the digestion process
- Full temperature control ensures complete digestion and higher safety

EXOTHERMAL REACTIONS

- Often exothermal reactions is noticed during the digestion of organic samples, such as:
  - Food, feed & beverage
  - Pharmaceutical
  - Biological
  - Petrochemical
- System has to be able to control the reaction to ensure:
  - High digestion quality
  - High safety of the process

MILESTONE easyTEMP

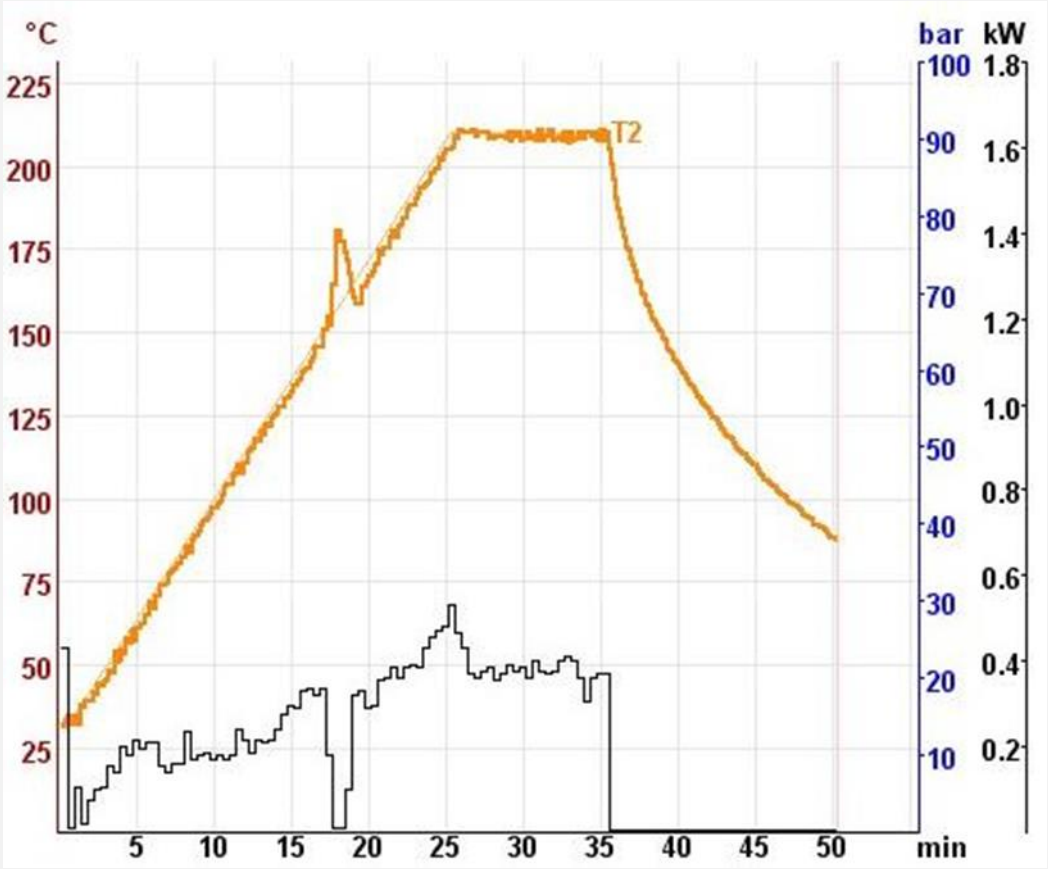
- Direct contactless sensor temperature control
- Temperature control in all vessels
- Quality control of the digestion process
- Complete data traceability
- Enhanced safety



easyTEMP CONTROL

- The combination of Milestone easyTEMP sensor and easyCONTROL software ensures full controls of the digestion process
- Integrated PID control
- Power is driven by the temperature control

Digestion of 0,5 g infant formula with 5 mL of HNO_3 65%



PERFORMANCE AND PRODUCTIVITY

- The digestion rotors directly impact the performance and the productivity in a digestion process
- The choice of the rotor has to take in account:
  - T & P required to achieve complete digestion
  - Productivity
  - Ease of use
  - Reliability of the components

SK-15
High Pressure
Rotor

MAXI-24 HP
High Performance &
Throughput Rotor

MAXI-44
High Throughput Rotor

VENT-AND-RESEAL TECHNOLOGY

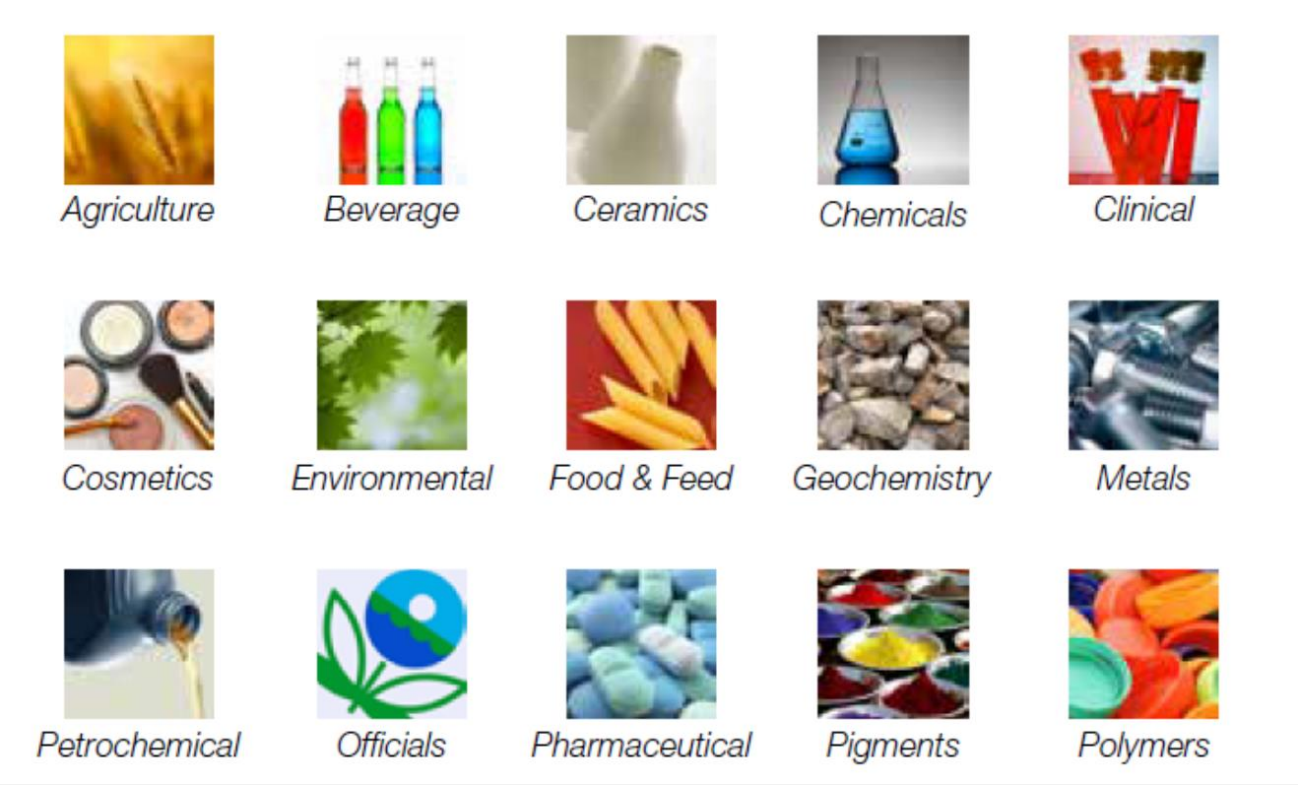
MILESTONE PATENT

- Reactive samples and high masses of organic sample, generate an increment of the internal pressure of vessels
- Vent-and-reseal is a safety mechanism to gently release the pressure only in case of overpressure
- This technology improves safety conditions by eliminating the need for a pressure sensor
- Vent-and-reseal ensures:
  - Full recovery of all elements
  - No loss of volatiles
  - High safety

MILESTONE SK-15

- High Pressure and Temperature rotor (260°C, 100 bar)
- 100 mL high purity PTFE vessels
- Suitable for :
  - Hard-to-digest sample
  - High sample amount
  - Highly reactive samples (exothermal reaction)

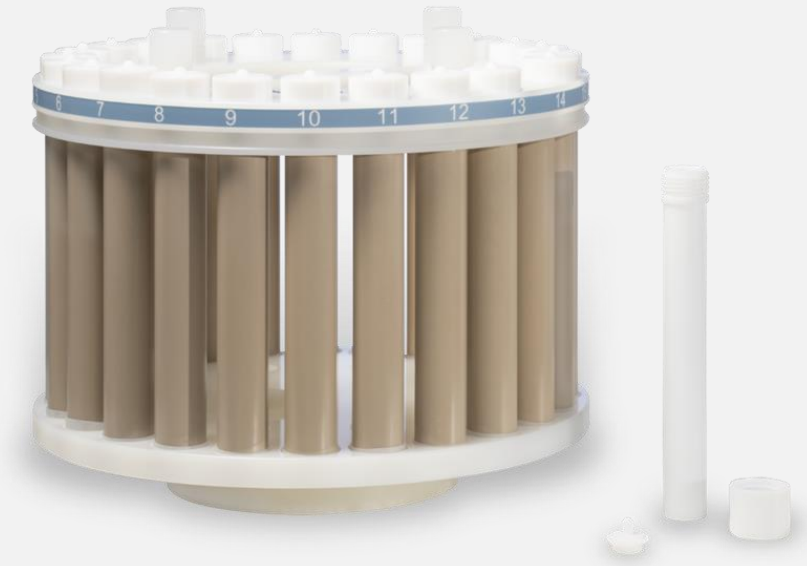
SK-15 TYPICAL APPLICATION



MILESTONE MAXI-24 HP

HIGH PERFORMANCE AND THROUGHPUT ROTOR

- High Performance and throughput (260°C, 60 bar)
- 80 mL high purity PTFE vessels
- Suitable for:
  - Moderate to high reactivity samples
  - Moderate to high sample amount
  - High throughput on several matrices

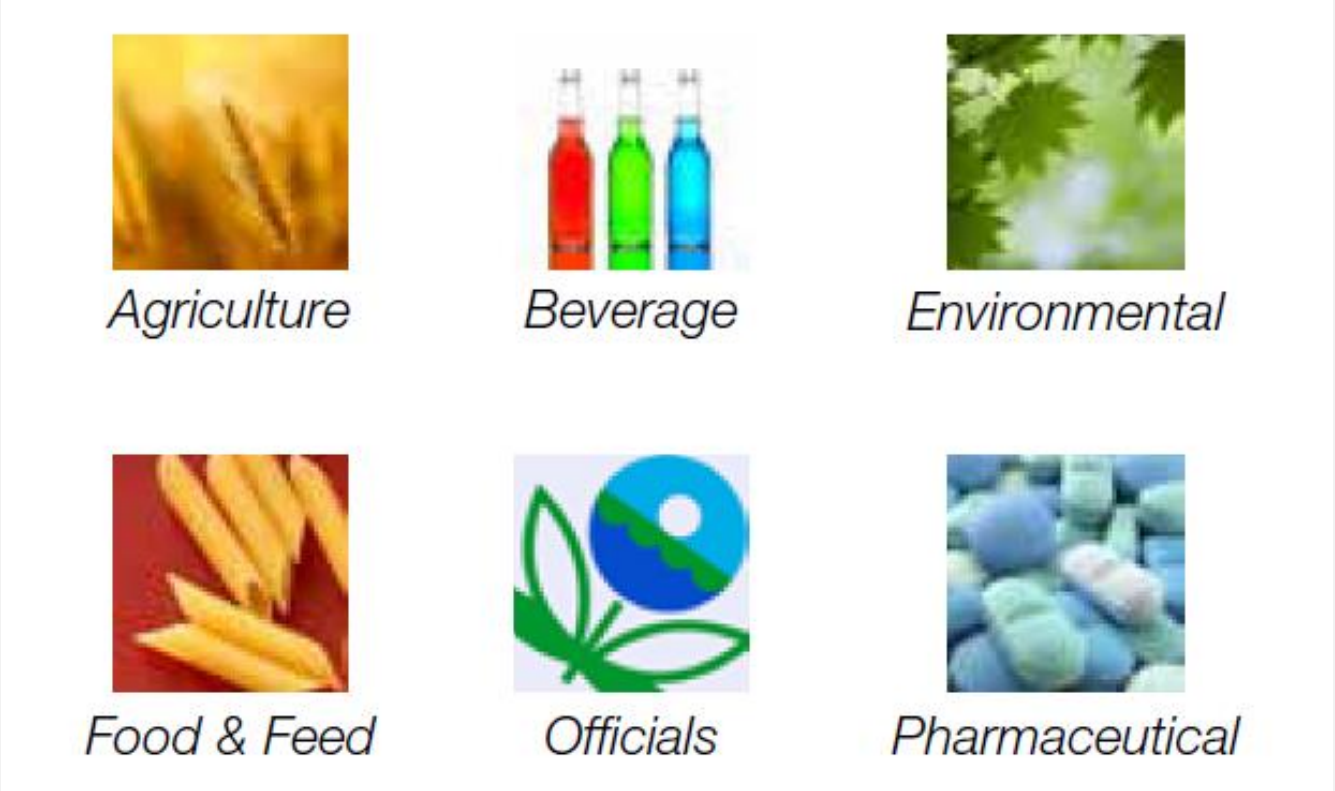


MAXI-24 HP TYPICAL APPLICATION

MILESTONE MAXI-44
HIGH THROUGHPUT ROTOR

- High Throughput rotor (260°C, 35 bar)
- 100 mL high purity PTFE vessels
- Suitable for:
  - Easy and medium-to-digest samples
  - Low reactivity (environmental samples)
  - Moderate sample amount

MAXI-44 TYPICAL APPLICATION



EXPERTISE AND KNOW-HOW

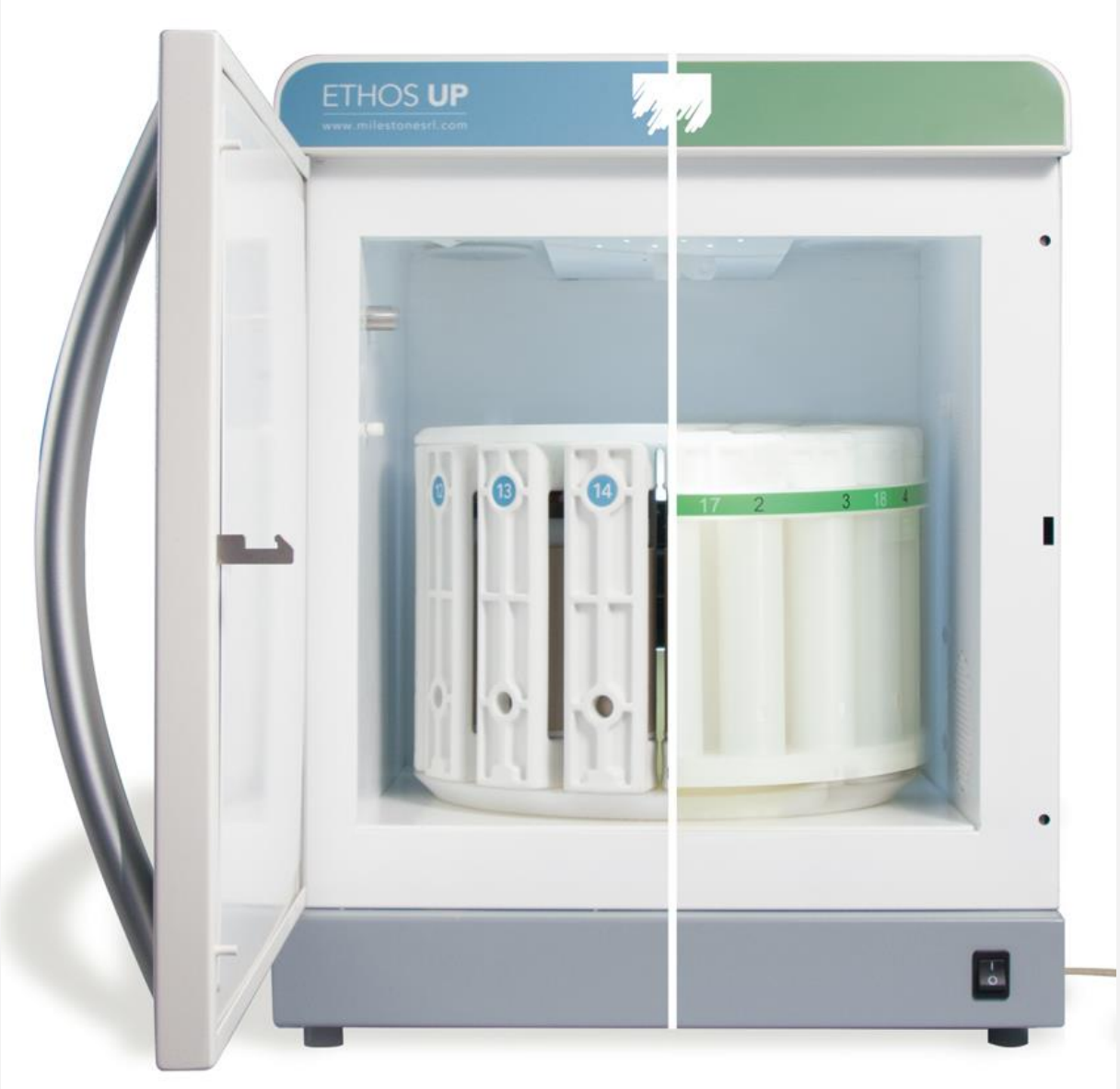
easyCONTROL SOFTWARE

- Icons-driven
- Multi-language
- 21CFR- Part 11 compliance
- Built-in application libraries
- More than 300 Application notes
- PDF creator

METHOD DEVELOPMENT

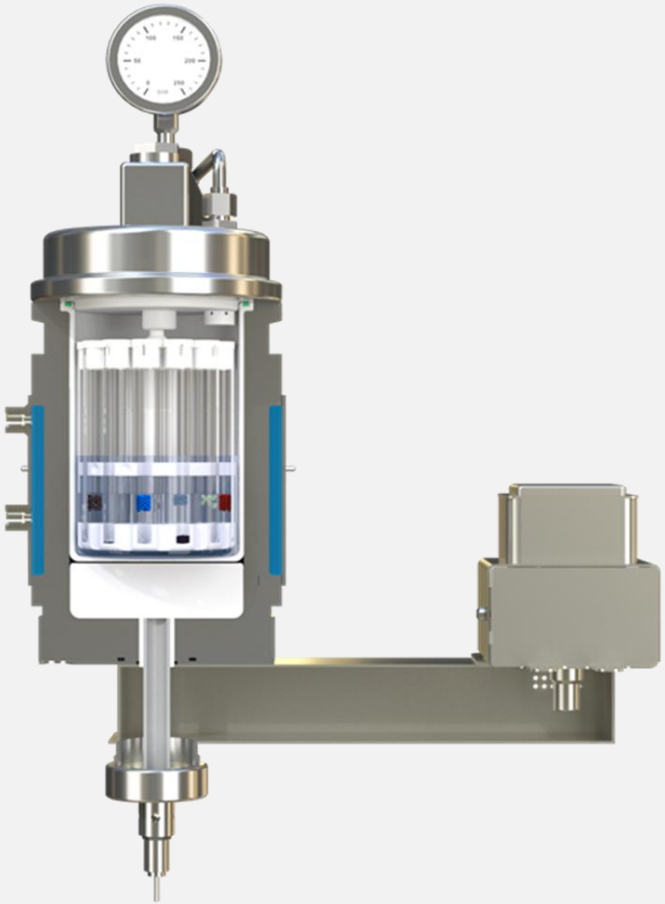
- Milestone library
  - Microwave programs
  - Sample weight and Acid mixture
- Milestone Connect
  - Application notes
  - Application reports
  - Scientific paper library
- Our experience at your service
  - Contact your local Milestone specialist

FLEXIBILITY



INTRODUCING THE SINGLE REACTION CHAMBER (SRC)

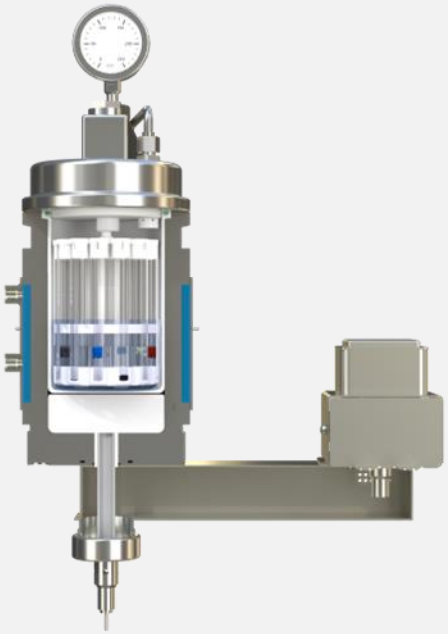
- SRC is a newer approach to microwave closed-vessel digestion
- Overcomes most limitations of rotor-based systems



HOW DOES IT WORK?

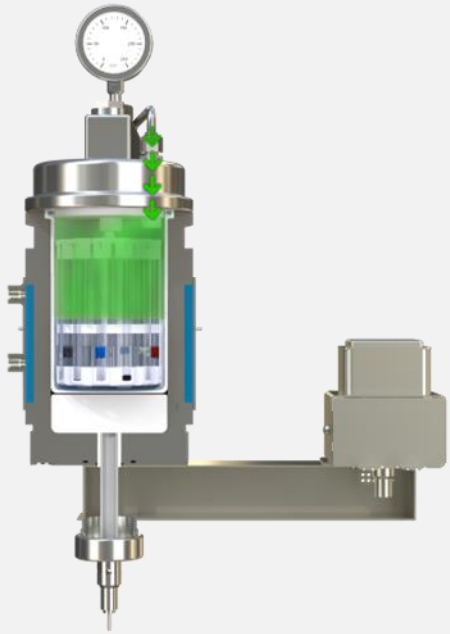
PRINCIPLE OF OPERATIONS

- Simple vials containing samples and acid are loaded into the chamber



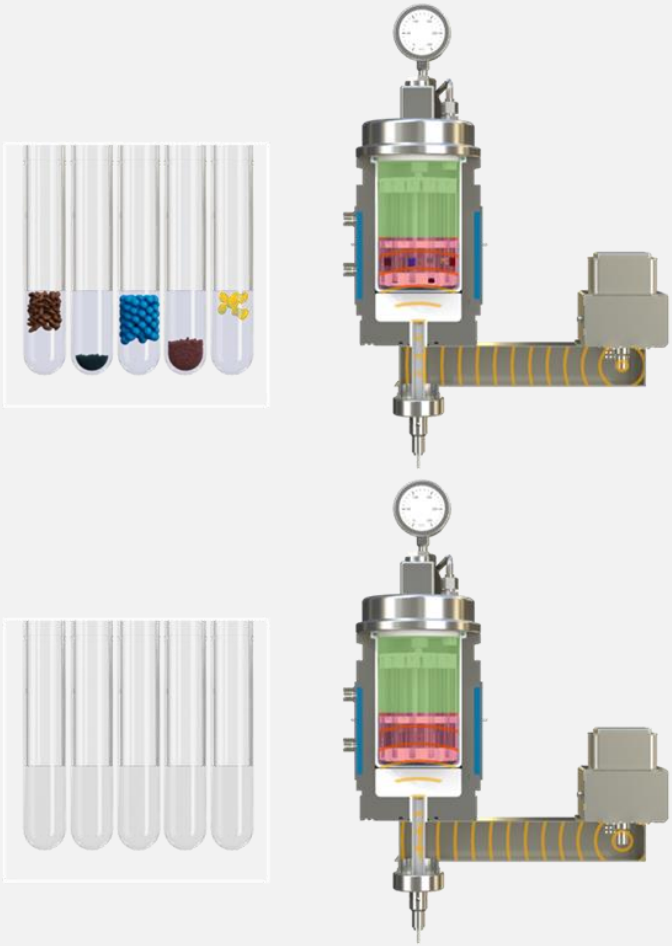
PRINCIPLE OF OPERATIONS

- Nitrogen is automatically loaded into the chamber



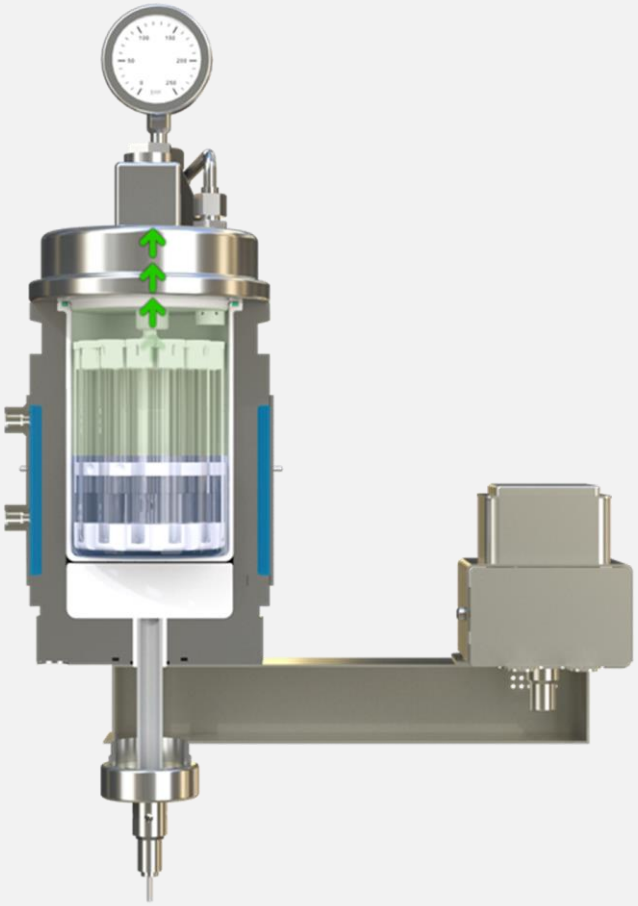
PRINCIPLE OF OPERATIONS

- Microwave program begins
- Sample digestion is completed



PRINCIPLE OF OPERATIONS

- Chamber is automatically cooled and vented



PRINCIPLE OF OPERATIONS

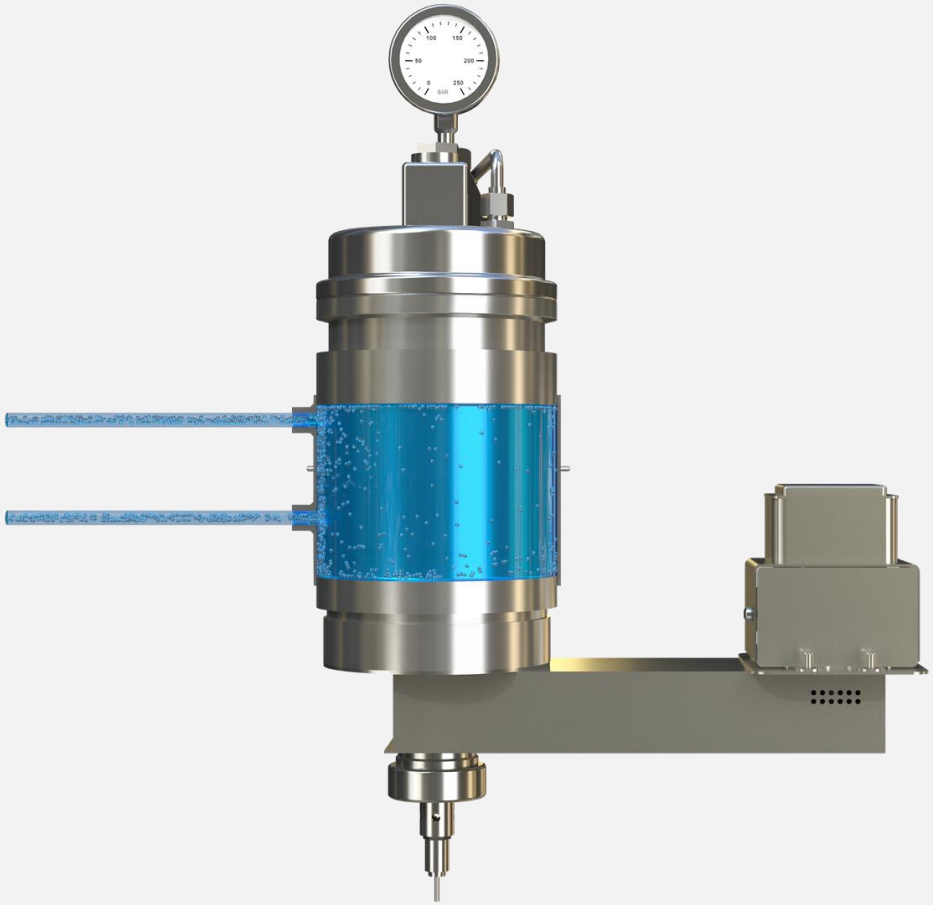
- Solutions are ready for dilution and analysis

TEMPERATURE AND PRESSURE IN THE SRC TECHNOLOGY

- SRC enables superior digestion conditions:
  - Up to 300°C
  - Up to 199 bar
- It leads to:
  - Higher digestion quality
  - Complete recovery (no venting, sample charring, or loss of volatiles)
  - Reduced interferences
  - Larger sample masses
  - Complete digestion of challenging samples

EFFICIENT HEATING AND COOLING

- Shorter digestion runs
  - Faster heating
  - Faster cooling by water chiller (12-14 min from 280°C to 80°C)
- Chamber always at low temperature
  - No acid diffusion
  - No overheating
- World’s first system with water-cooled magnetron
  - Superior heating efficiency
  - Longer lifetime
  - Noiseless operation



ULTRAWAVE 3 CONSTRUCTION

- High-pressure lines made of:
  - Corrosion-resistant steel
- Separated high-pressure lines:
  - Inlet for gas pre-pressurization
  - Outlet for venting at the end of the run
    - Lower blanks
    - Longer lifetime of the lines

HIGHEST PRODUCTIVITY

- Number of samples per run
- Multiple sample types simultaneously
- Cleaning time

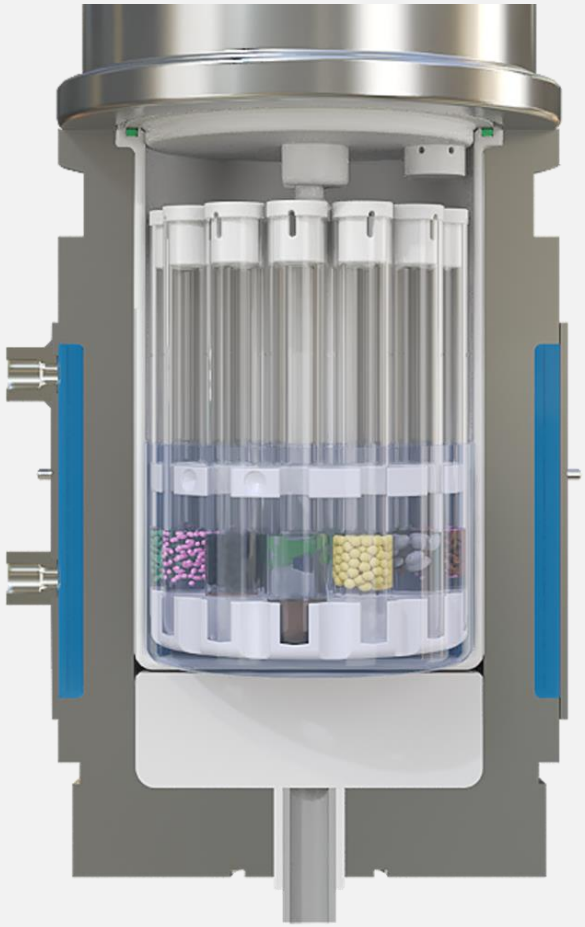
RACKS AND VIALS

- Inexpensive construction
- Vials available in:
  - PTFE-TFM
  - Quartz
  - Disposable Glass

# of position	Volume (mL)	Vials material
7	40	PTFE-TFM, Quartz, Disposable glass
20	15	
27	8	
40	4.5	Glass

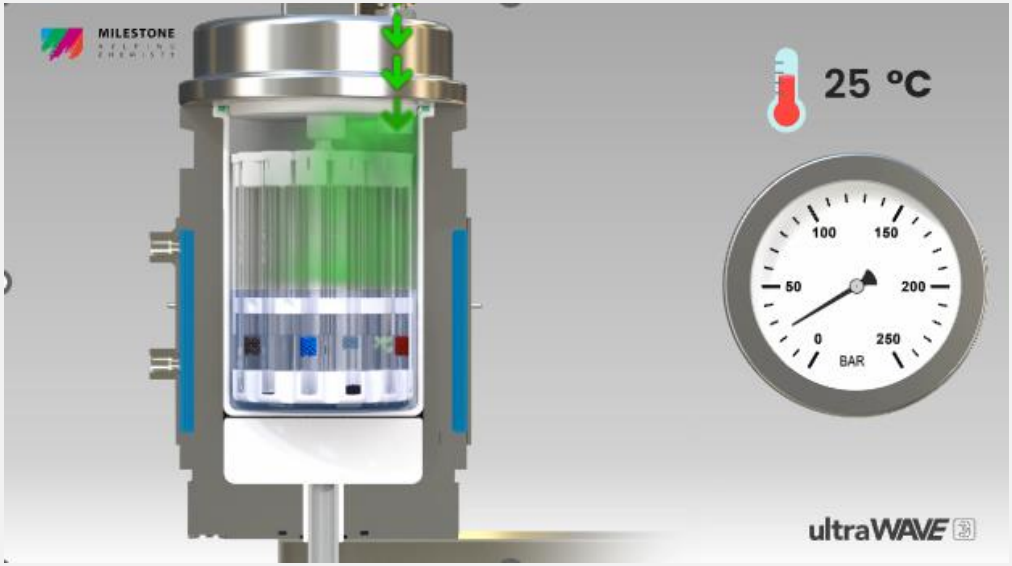
SAME TEMPERATURE IN ALL VIALS

- Rack with vials sits in a water base load
  - Homogenous heating
  - Homogenous temperature
  - Optimum heat dissipation in case of exothermal reaction

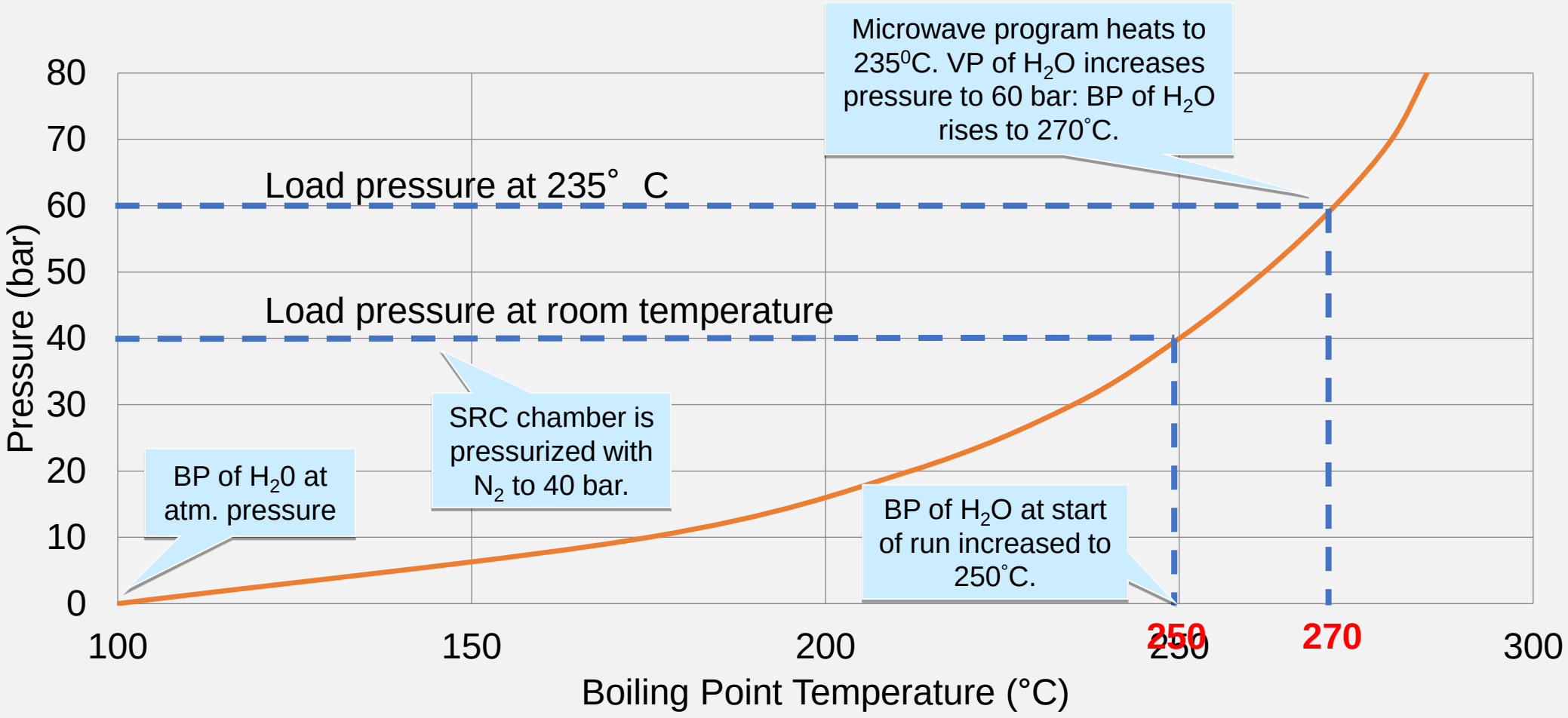


SAME PRESSURE THROUGHOUT THE ENTIRE CHAMBER

- Pre-pressurization with nitrogen
  - Ensures all the analytes stay in solution
  - Avoids reaching the boiling points of the acids
  - No charring
  - Pressure equilibrium between the chamber and the vials



H_2O BOILING POINT TEMPERATURE VS. PRESSURE



ALL SAMPLE AT ONCE

Any sample, any chemistry, any volume: IN A SINGLE RUN!

Questions!