

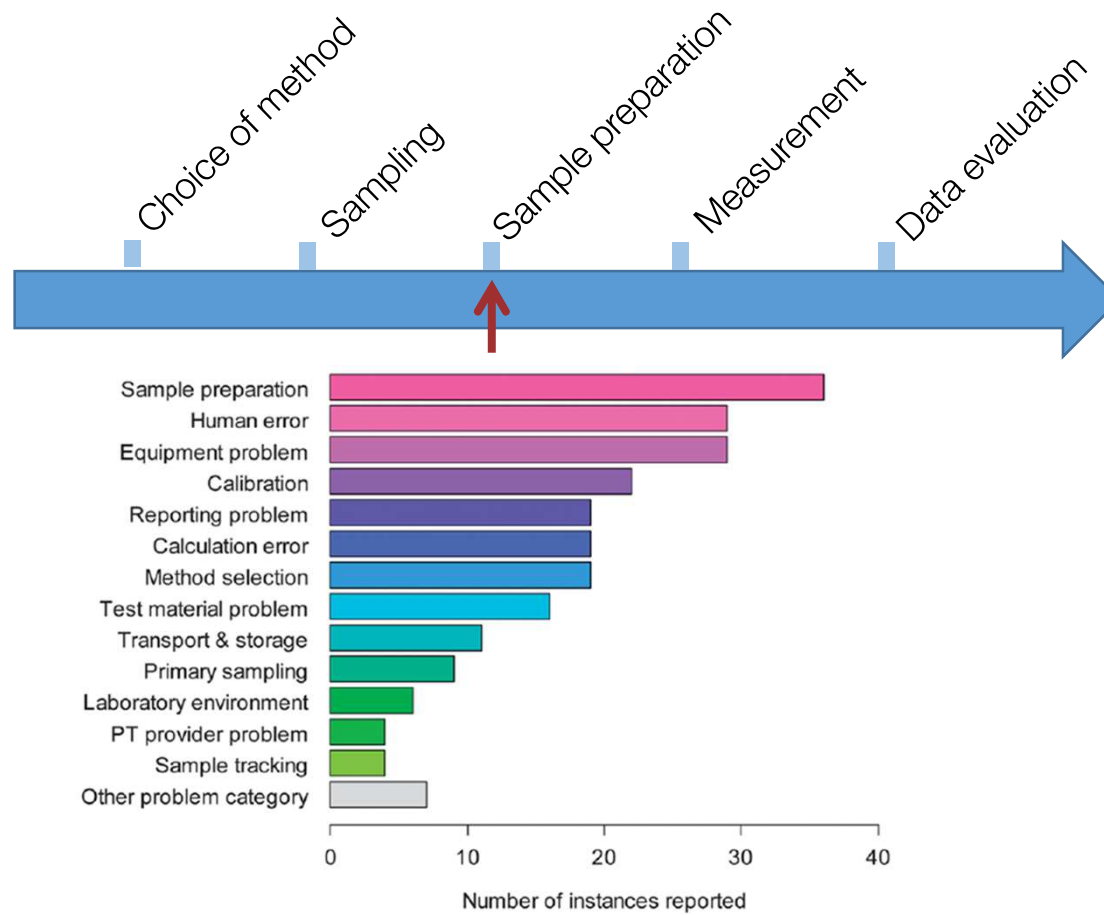
The best combination of sample preparation and elemental analysis technologies for controlling the analytical blank

Milestone srl



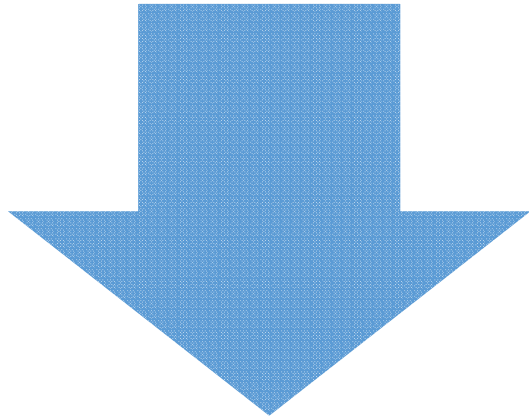
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The analytical sequence



Analytical Methods, 5:2914–2915, 2013.

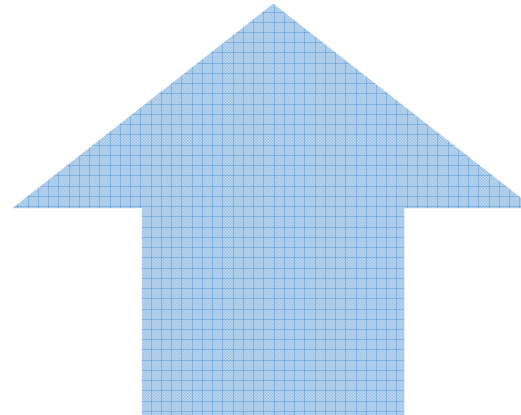
Sample prep is ...



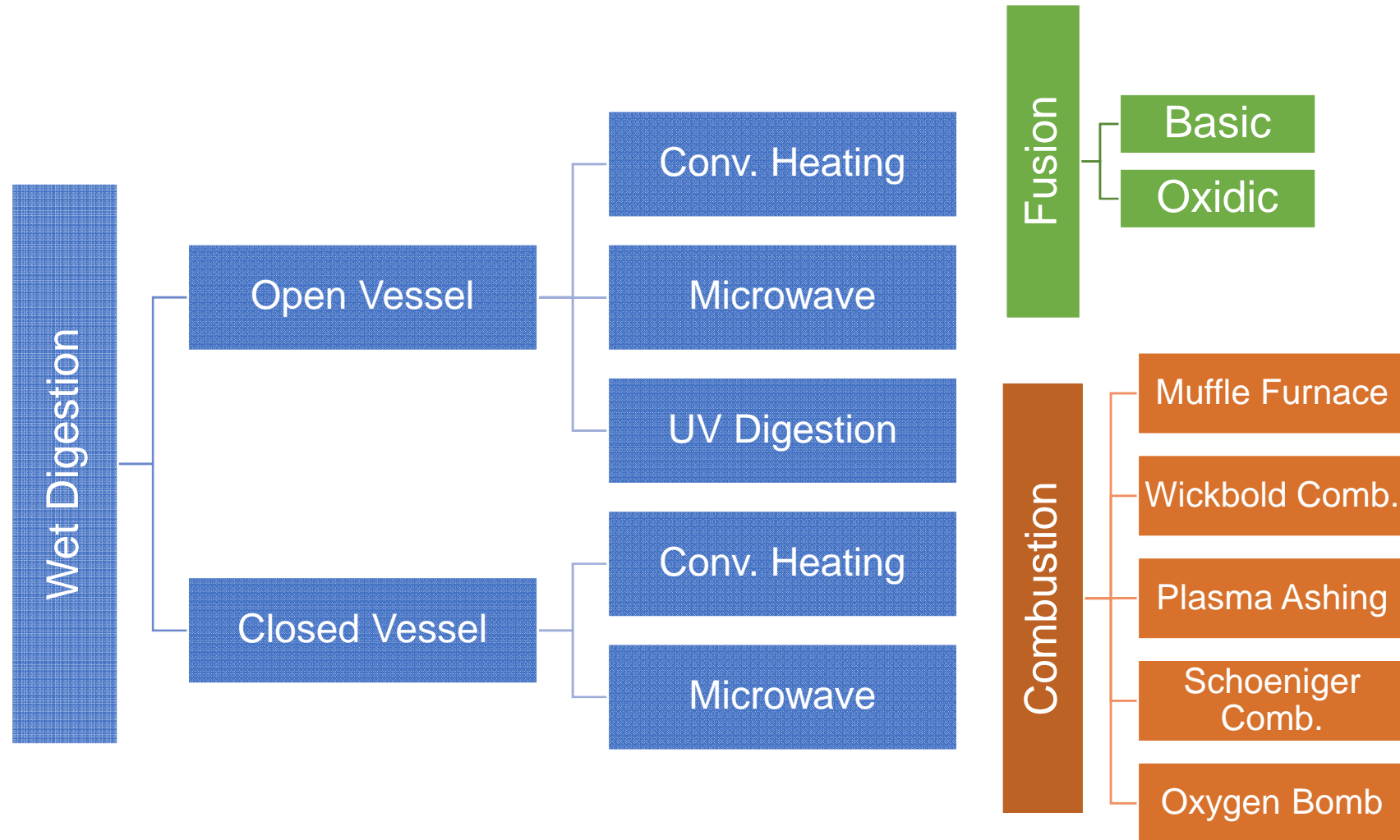
...the
bottleneck in
the
laboratory



...the key to
accurate
analysis



Sample prep Methods



Microwave Closed Vessel Technology

Will likely be the most common closed-vessel digestion technique due to:

- Established technology in sample prep
- High digestion quality
- No losses of volatile elements,
- Low blanks
- High Safety
- High throughput / productivity
- Easy of use



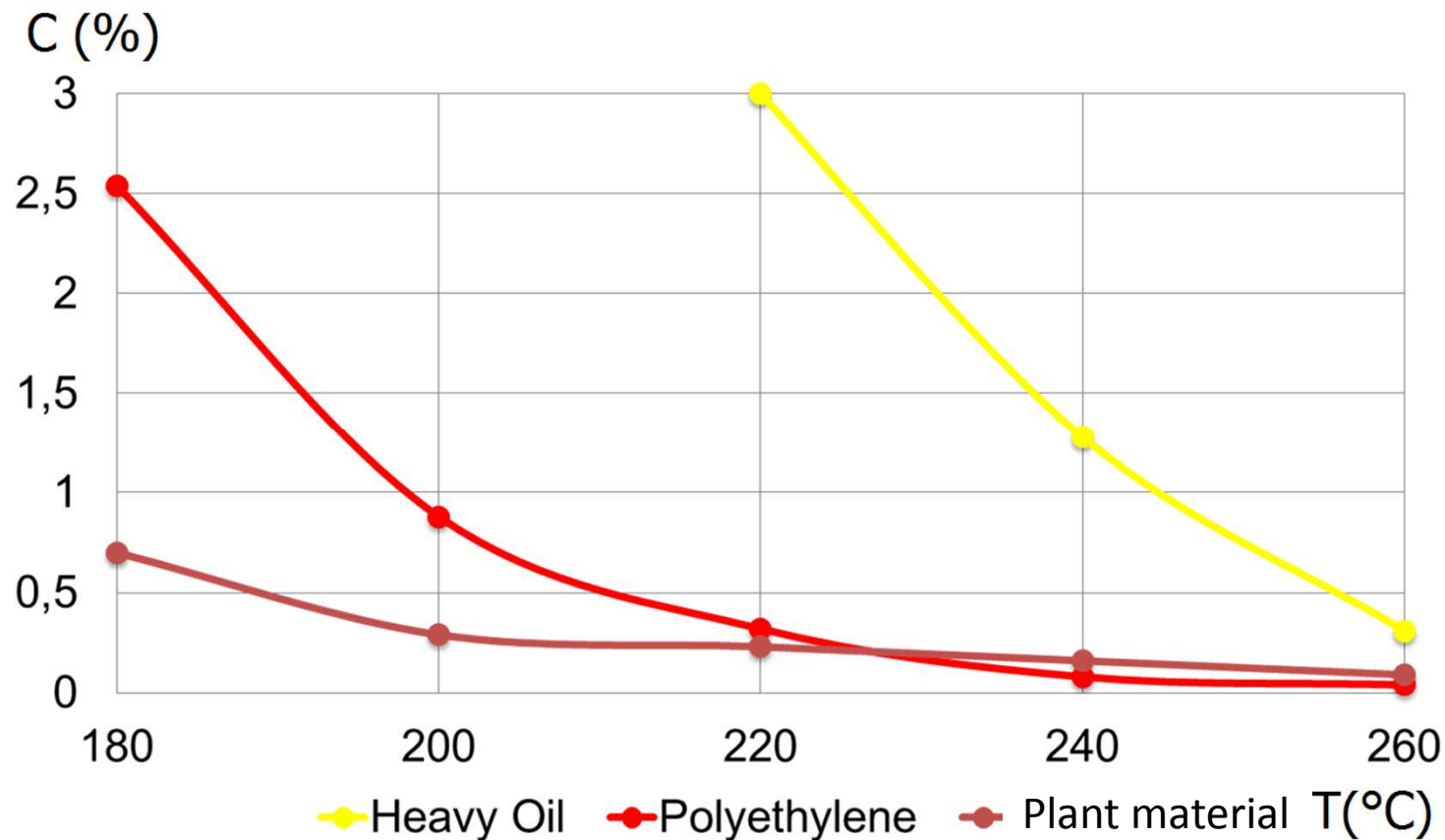
Microwave digestion sample prep enables more accurate and more sensitive trace metal analysis, over a much wider range of sample types

Key parameters to select the right microwave digestion system

- **Sample type/amount** The choice of the right configuration strongly depends on your samples and your analysis technique.
- **Productivity** Number of samples per day/ week
- **Ease of use** Handling time, easy to work with
- **Flexibility** Capability to be used with any number of vessels and different configurations
- **Reliability** Robustness, hardware construction
- **Rotors/ vessels must be able to handle high T and P conditions**

Digestion Quality

Residual Carbon Content vs Digestion Temperature



Lower carbon residue results from higher digestion quality. High carbon content generates interferences and clogging in the ICP-OES / ICP-MS

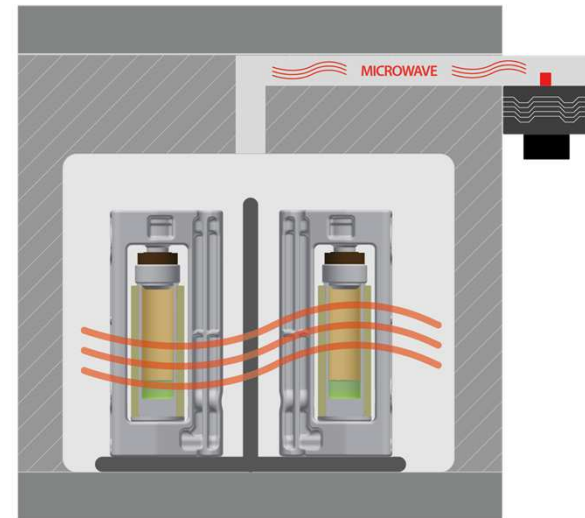
Conventional closed vessel Microwave Digestion

Advantages

- High pressure, high temperature digestion
- Low acid use, so low reagent blanks
- No loss of volatile elements
- Low risk of contamination

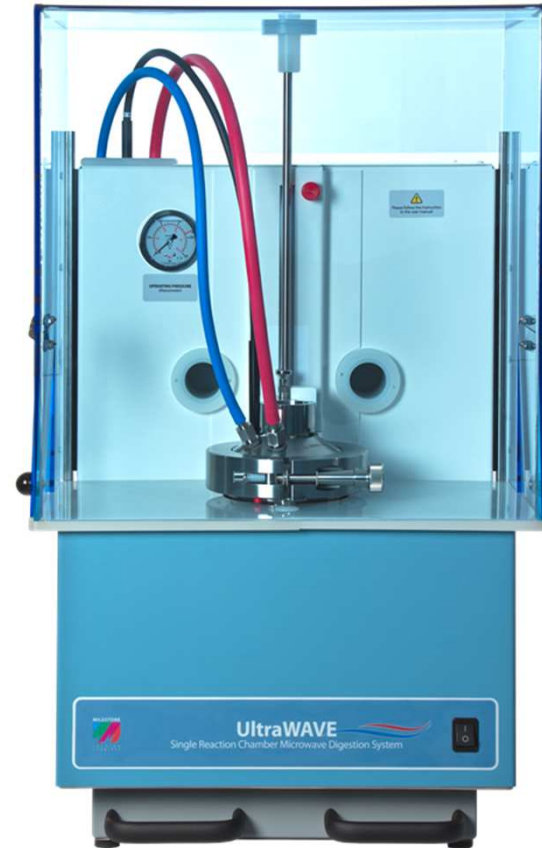
Limitations

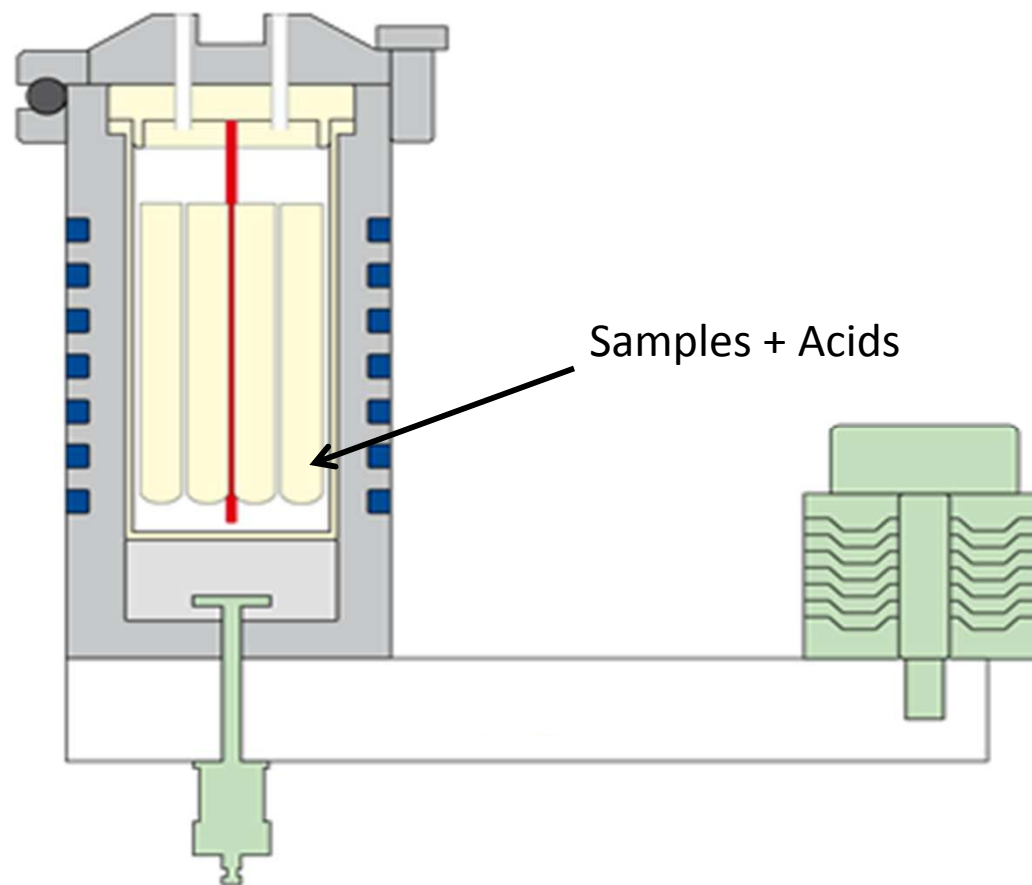
- Vessel capping/uncapping and cleaning
- Must batch into samples of same type
- Higher consumables cost



SRC Technology: UltraWAVE

- **Highest Temperature and Pressure Available**
 - Working temperature: up to 300°C
 - Maximum pressure capability: up to 200 bar
 - Digest even the most difficult/stable samples
- **Digest completely mixed sample matrices simultaneously**
 - One run for all sample type
- **Easy handling**
 - No assembly/disassembly of vessels
 - No cleaning- ability to use disposable vials
- **Extreme Productivity**
 - High sample throughput
 - One digestion program for everything
- **Lower operating costs**
 - Reduced labor and consumables costs





Direct pressure and temperature control of the chamber –
Equilibrium of pressure and temperature between all vials



Racks and Vial Types

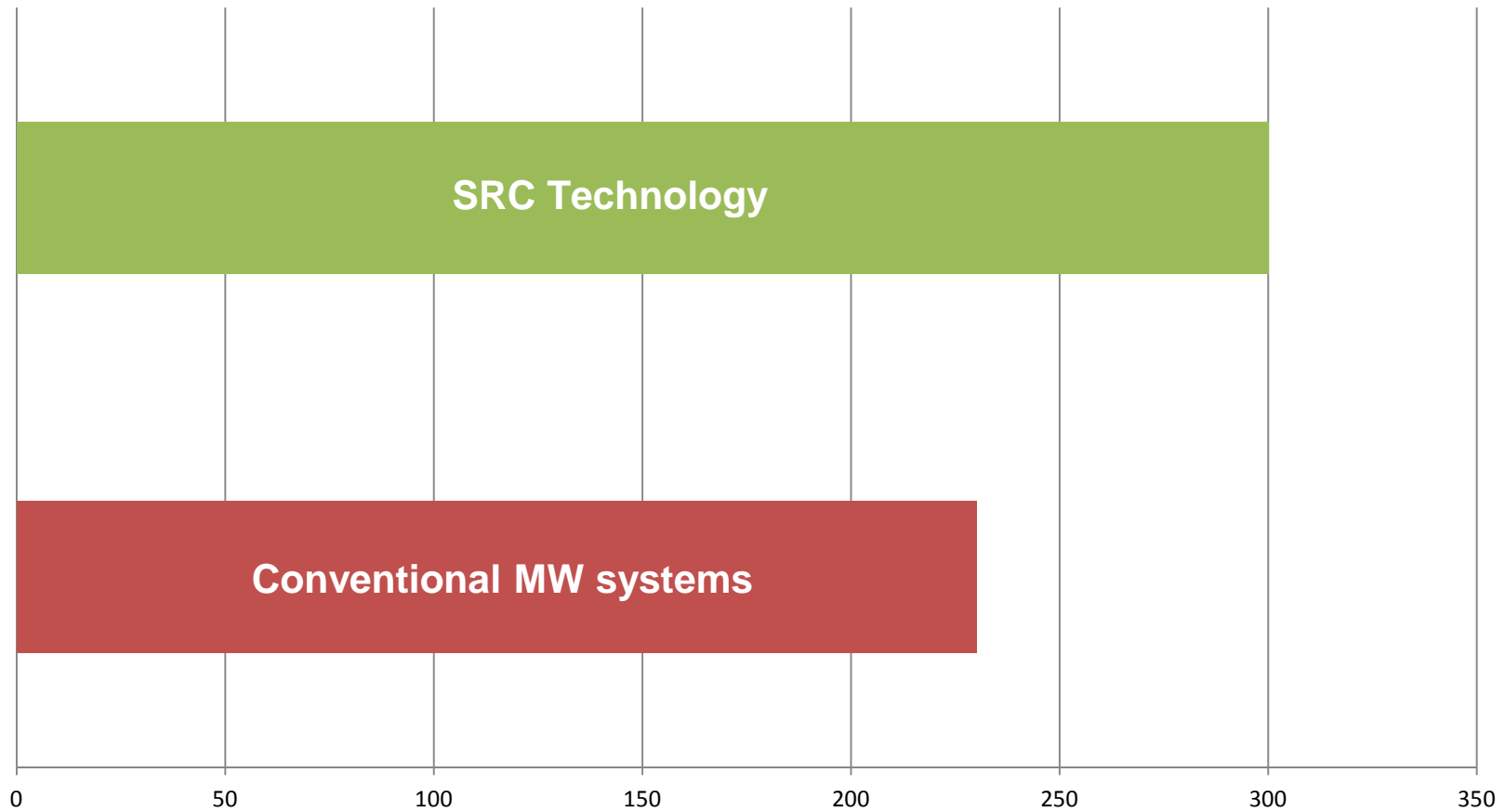
- Variety of sample rack sizes 4, 5, 15, 22 position racks.
- Three vial types
 - Borosilicate glass (disposable)
 - Quartz
 - TFM (applications needing HF)
- Loose-fitting TFM caps



Microwave technologies at comparison

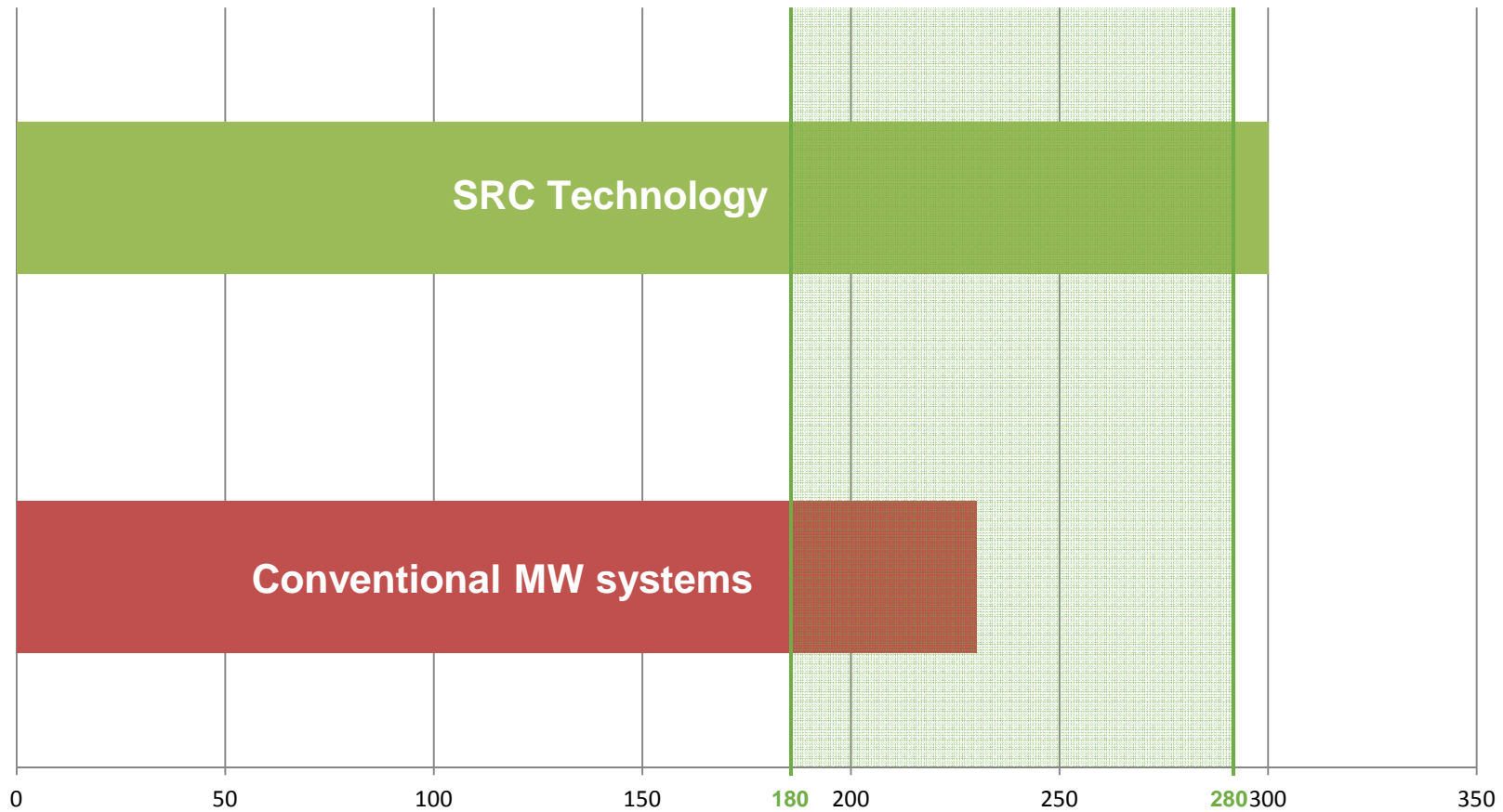
	Conventional MW systems	SRC technology UltraWAVE
Working temperature with an organic sample	Up to 230°C	300°C
Maximum pressure capability	Up to 100 bar	200 bar
Number of samples per run	15 vessels per run with high pressure	15 vials per run
Typical Digestion Time (including cooling time)	1h and 30 min for 15 vessels	Less than 1h for 15 vials
Productivity	Medium	High

Temperature performances (°C)



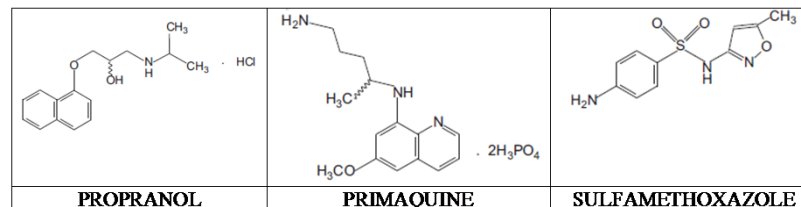
Temperature performances (°C)

Most of organic and inorganic samples require a digestion temperature between 180°C - 280°C

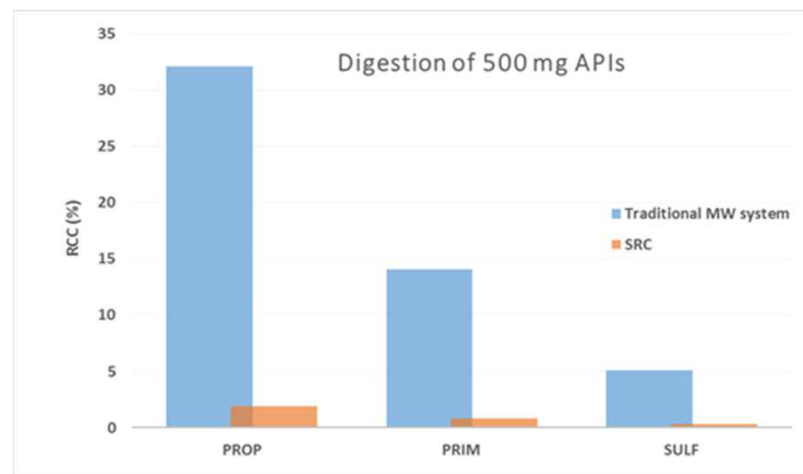


High efficient digestion over the traditional microwave equipment

- UltraWAVE achieved 270°C of digestion temperature with HNO₃



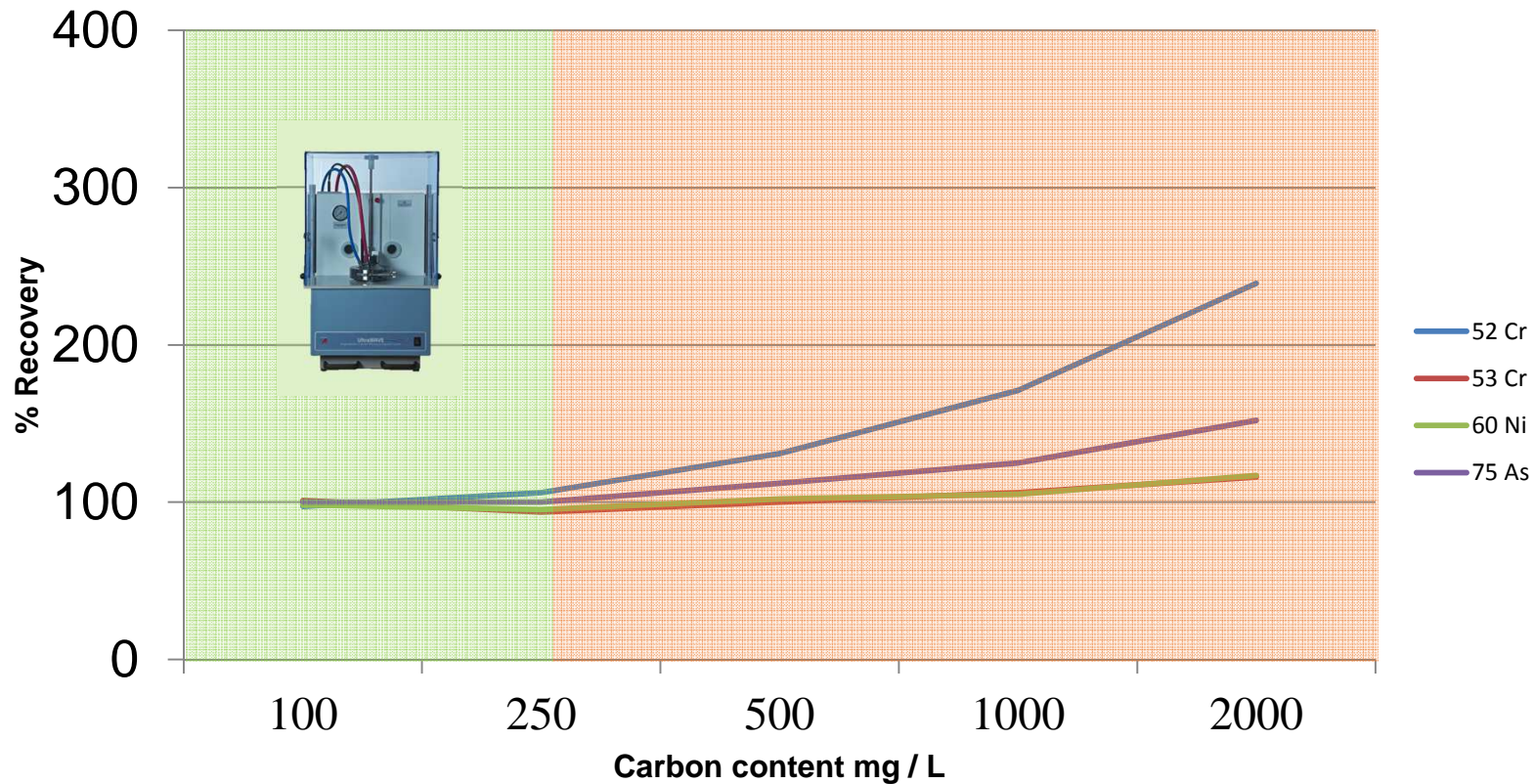
- SRC (UltraWAVE) ensures a very low residual C content (lower than 250 mg L⁻¹) that results in a perfect recovery.



- Less carbon interferences

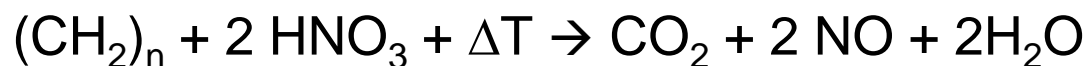
“Study and determination of elemental impurities by ICP-MS in active pharmaceutical ingredients using single reaction chamber digestion in compliance with USP requirements” A.L.H.Muller, J.S.S.Oliveira, P.A.Mello, E.Muller, E.M.M.Flores - Talanta136(2015)161–169

The Effect of the Carbon residue on the ICP-MS analysis



Basic Considerations

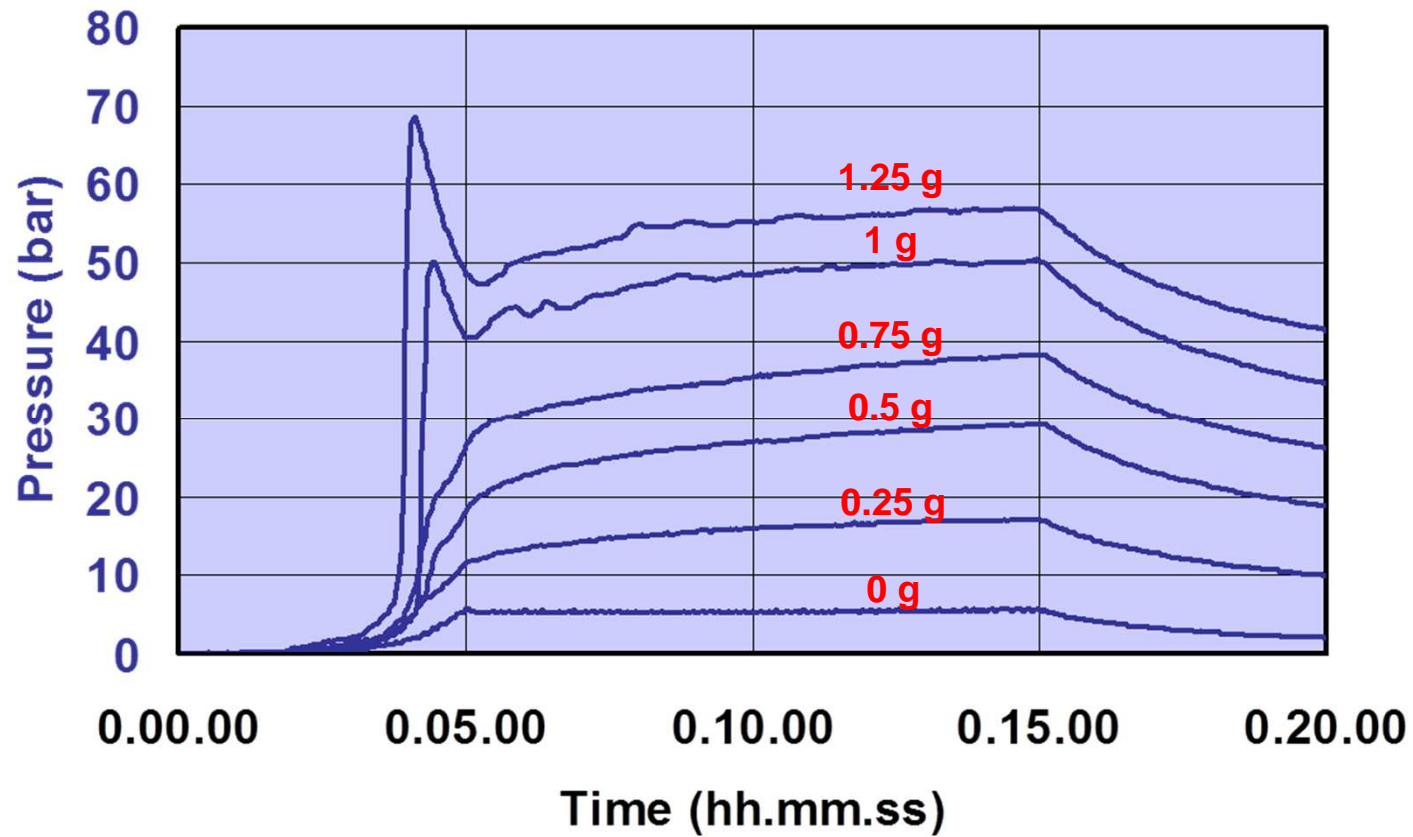
- Reaction equation



- More sample \rightarrow more gas \rightarrow higher pressure
- Vessel and microwave system have to be capable of withstand or handle high temperatures and pressure conditions
- High temperature capabilities in a vessel with low pressure limit will not allow to achieve good digestion

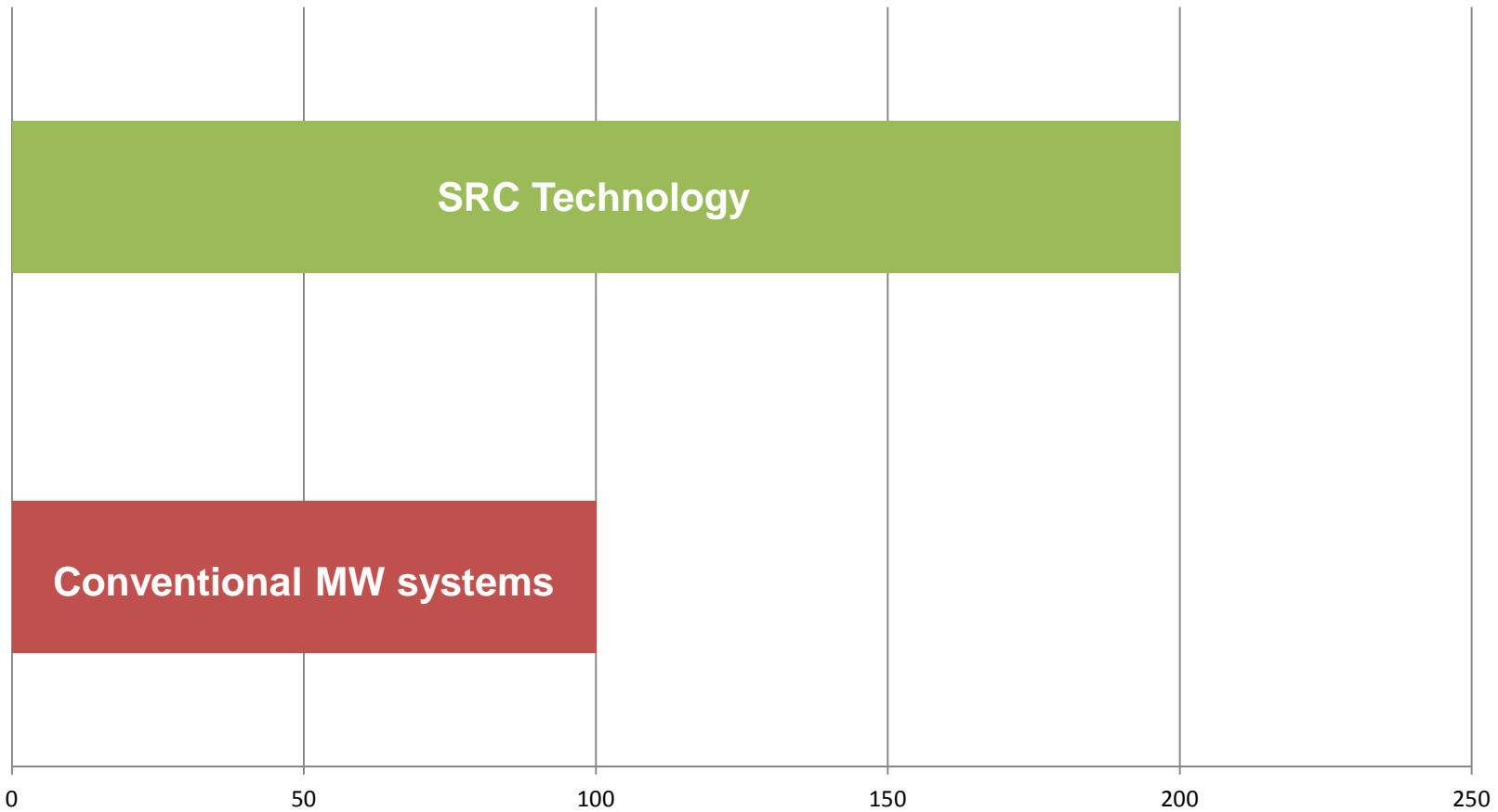


Effect of Sample Amount on Pressure



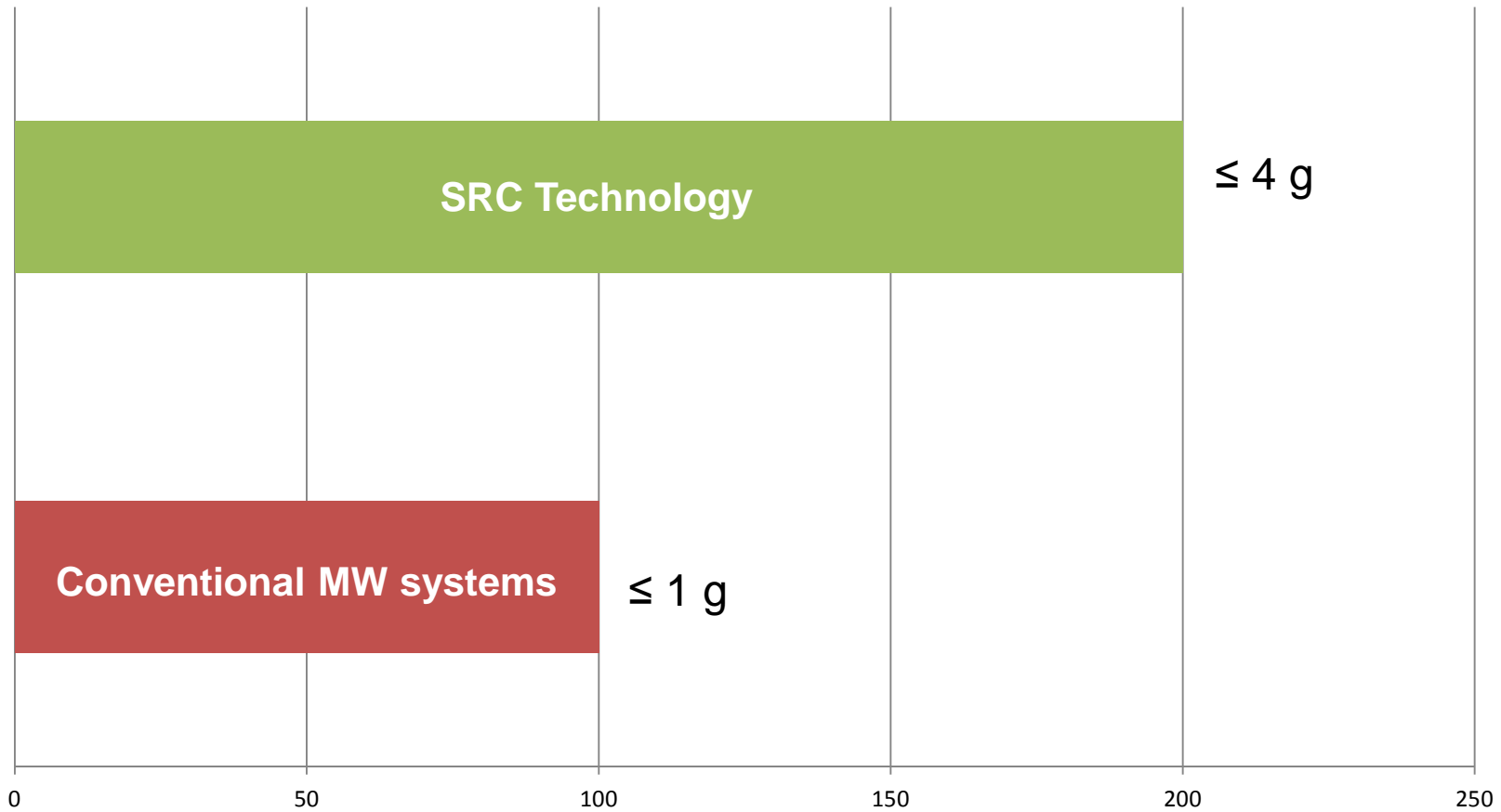
Pressure performances (bar)

Sample amount



Pressure performances (bar)

Sample amount



Combination of high Pressure and Temperature for sample prep

- Higher digestion quality
 - Less interferences
 - Lower residual carbon improves the ICP's stability
 - Lower ICP maintenance
- Higher sample amount
 - Improved LOQ
 - Great for trace metal determination.
 - Better sample homogeneity
- Simplified digestion process
 - Easy handling
 - Mixed samples in one run
 - Higher productivity
 - Less acid consumption



Thank you for the
attention!



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