



Optimization of a Microwave digestion - ICP-AES method for foliage samples

Alfred FÜRST

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Situation in the lab

- 5000 foliage samples/year on ICP-AES
- Parameters
 - Macronutrients P, K, Ca, Mg
 - Micronutrients Fe, Mn, Zn, B, Cu
- Contamination!
- Losses (fixing of Fe on the silica matrix)!
- Blocking of the ICP-AES Nebulizer (silica precipitation)!

Microwave digestion

CEM – Mars 6

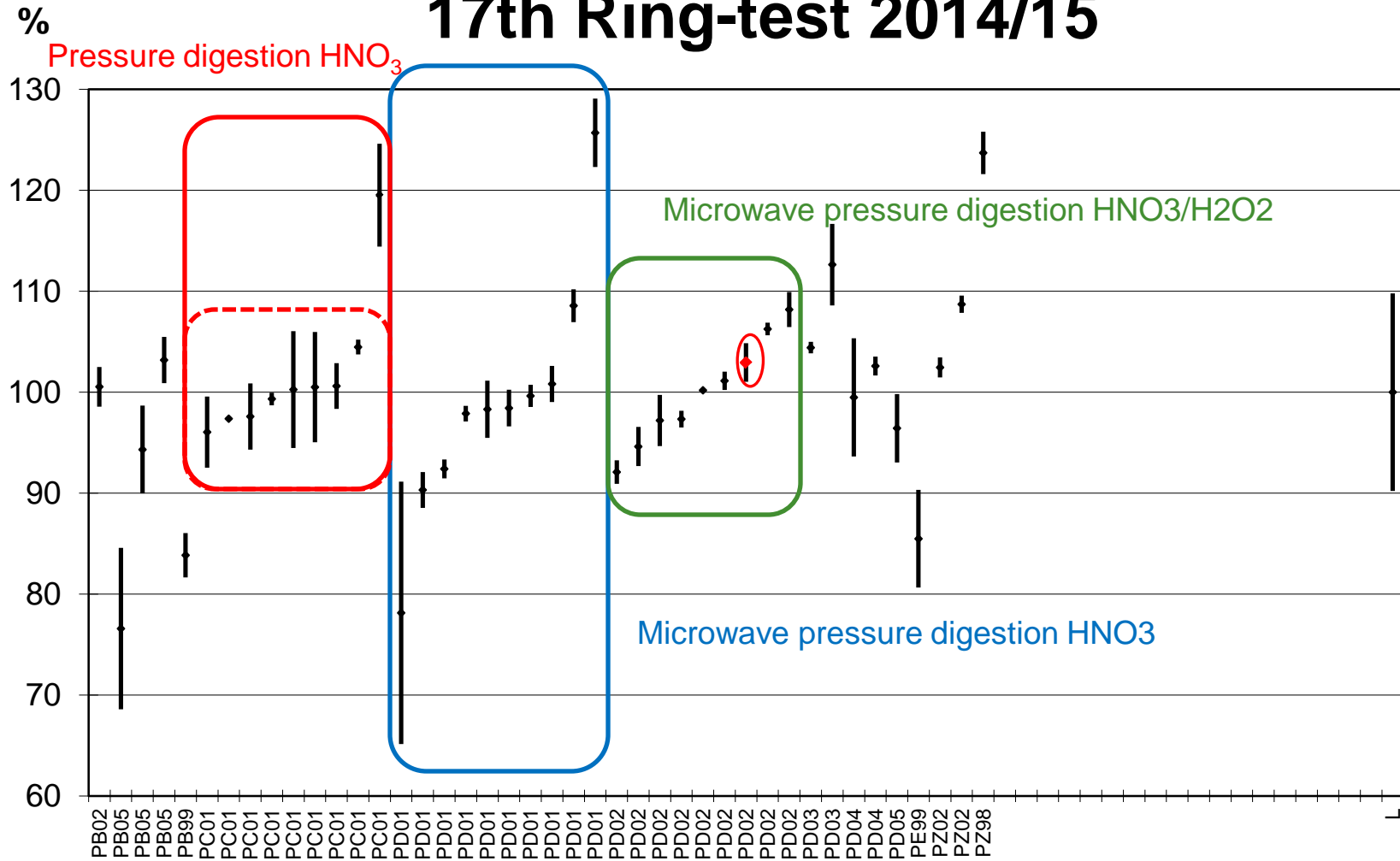
- **40 samples / run**
- Temperature control of each sample
- End temperature **200° C** for **15 min.**
- **Contamination free** teflon vessels
- 3 separate trays with 120 vessels
- 80-120 samples/day



Microwave digestion

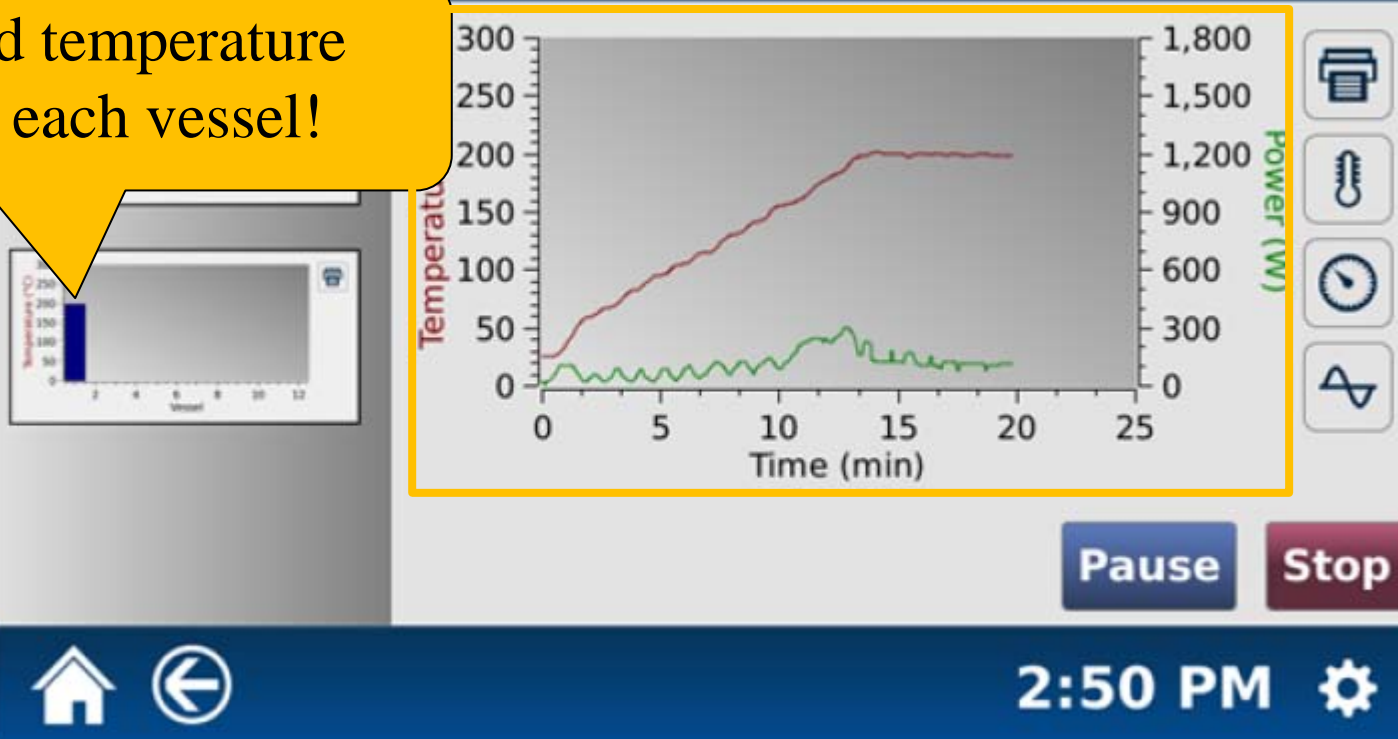
- 250 – 350 mg needle (foliage) sample
- Add 5 ml nitric acid
- Shake carefully
- Put the plug on the vessel but leave it uncapped; stand over night
- Add 1 ml H₂O₂
- Cap the vessel and put it in the liner
- Heat it up in the microwave (15 min. ramp to **200° C**; 15 min. hold on 200° C)

Pretreatment Recovery Fe-Sample 1 17th Ring-test 2014/15



Run - Plant Material

End temperature
for each vessel!



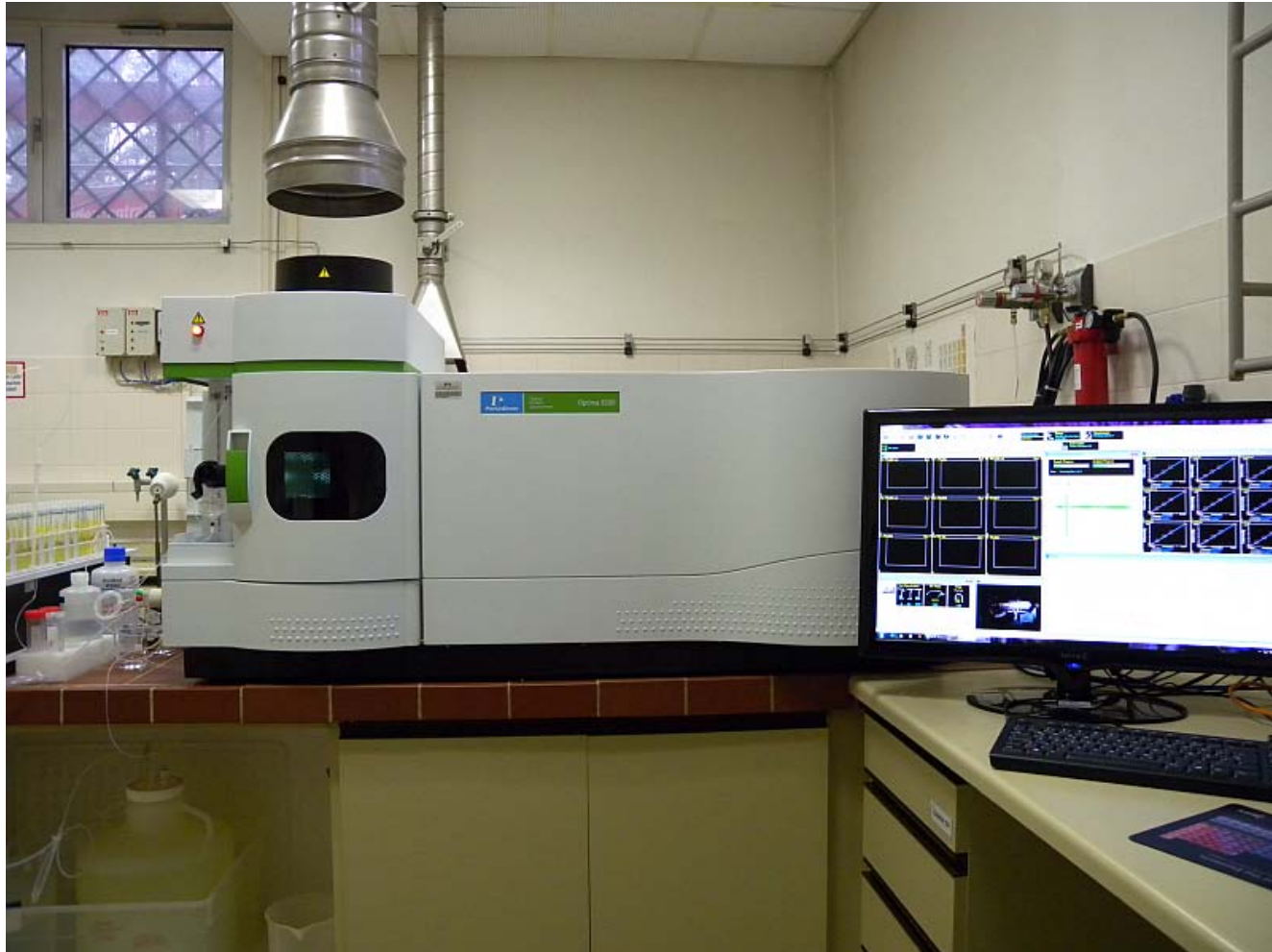
Temperature/Power/Time Graph

Sample preparation

- Cool down the vessels (30 min.)
- Uncap the vessels
- Add 44 ml deionized water (~ 50 ml total)
(Contamination risk: **No fillig up in a volumetric flask!**)
- Put the total sample in a PE-Flask for storing till measurement
- Silica particles should sedimented
(Contamination risk: **No filtration!**)

ICP-AES

PE-Optima 8300 DV + Fast Sampler



Premixed Standard Solutions

Element	Konzentration
K	1000 mg/l
Ca	1000 mg/l
Mg	500 mg/l
Mn	200 mg/l
Fe	50 mg/l
P	500 mg/l
Zn	20 mg/l
Cu	2 mg/l
B	20 mg/l
Matrix	5% HNO ₃

Certificate of Analysis

CERTIFIED REFERENCE MATERIAL

Multi-element Standard Solution

MiraMist-Nebulizer

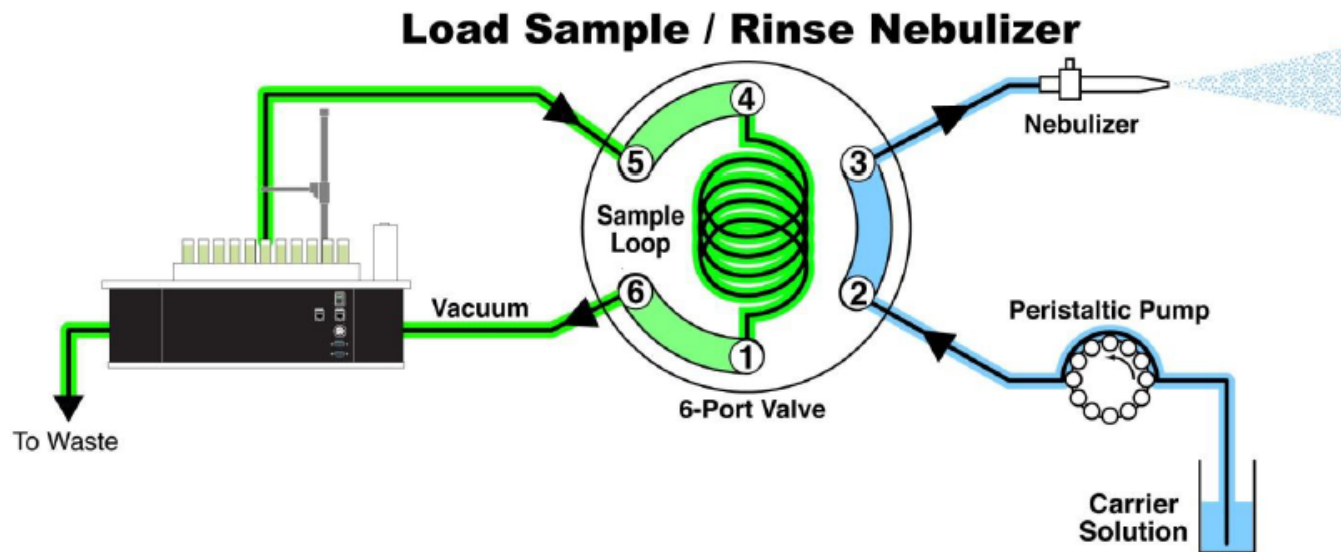
- **Can handle high level of particulates** in sample (only a hole with the same diameter like the sample tube)
- Low sample flow (0.2 ml/min to 2.5 ml/min)
- Standard pressure (45 psi)



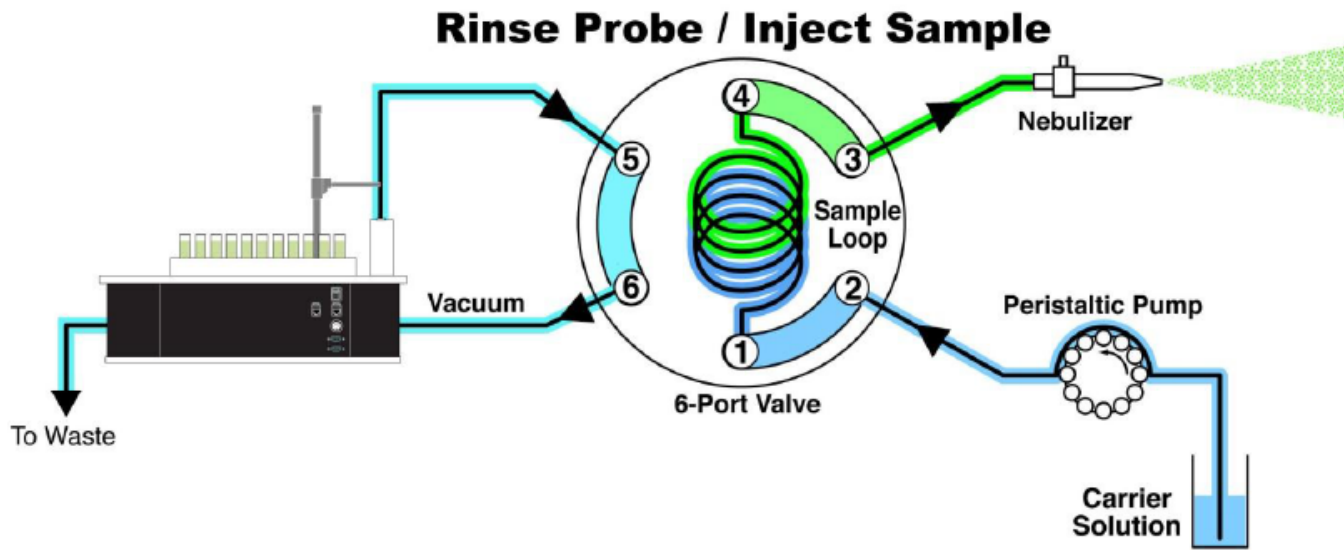
Fast Autosampler

Contamination free
teflon sample loop, teflon
tubes and spraychamber!





Fast filling of the sample loop (5-7 s)



Sample loop with 1.5 - 2 ml
 Low amount of sample is transported in the plasma
 No memory effects (e.g. Zn, B) from the peristaltic tubes