

# MULTIPARAMETRIC MICROLOOP FLOW ANALYSER WITH MINIATURIZED ION SELECTIVE ELECTRODES FOR QUALITY ANALYSIS OF CHEMICAL PARAMETERS



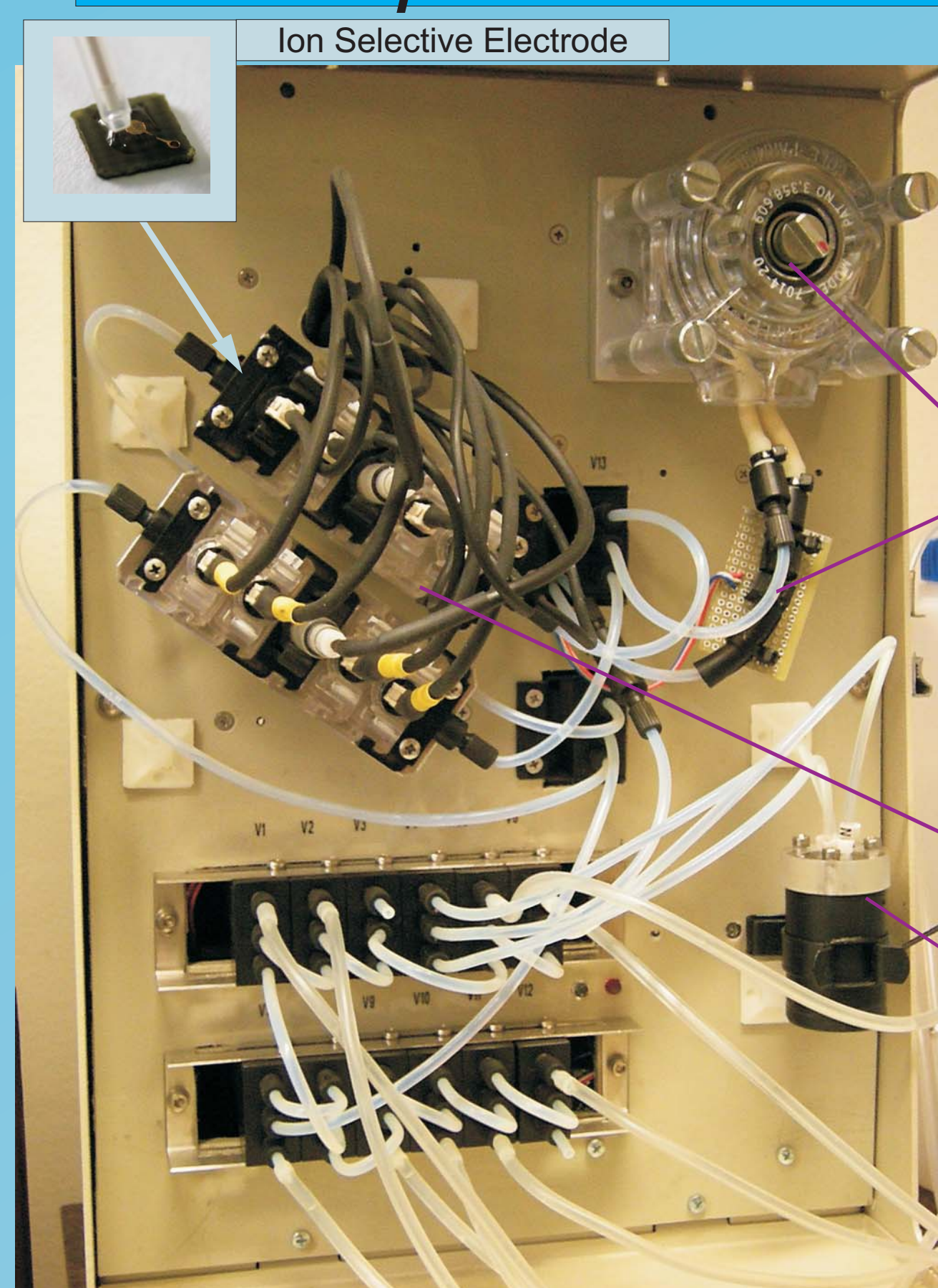
L.J. Opalski \*, P.Z. Wiczorek \*, P. Moscetta \*\*, L. Sanfilippo \*\* M. Malizia \*\*\*



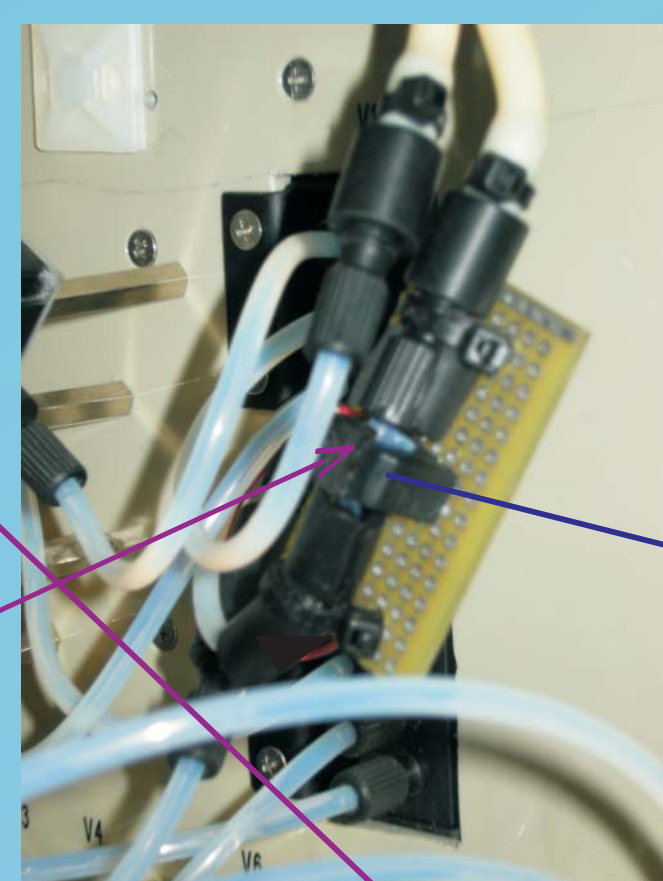
\*Institute of Electronic Systems, Warsaw University of Technology (WUT), Poland  
 \*\*Systema S.p.A., Anagni, Italy, \*\*\*Sysmedia S.r.l., Anagni, Italy



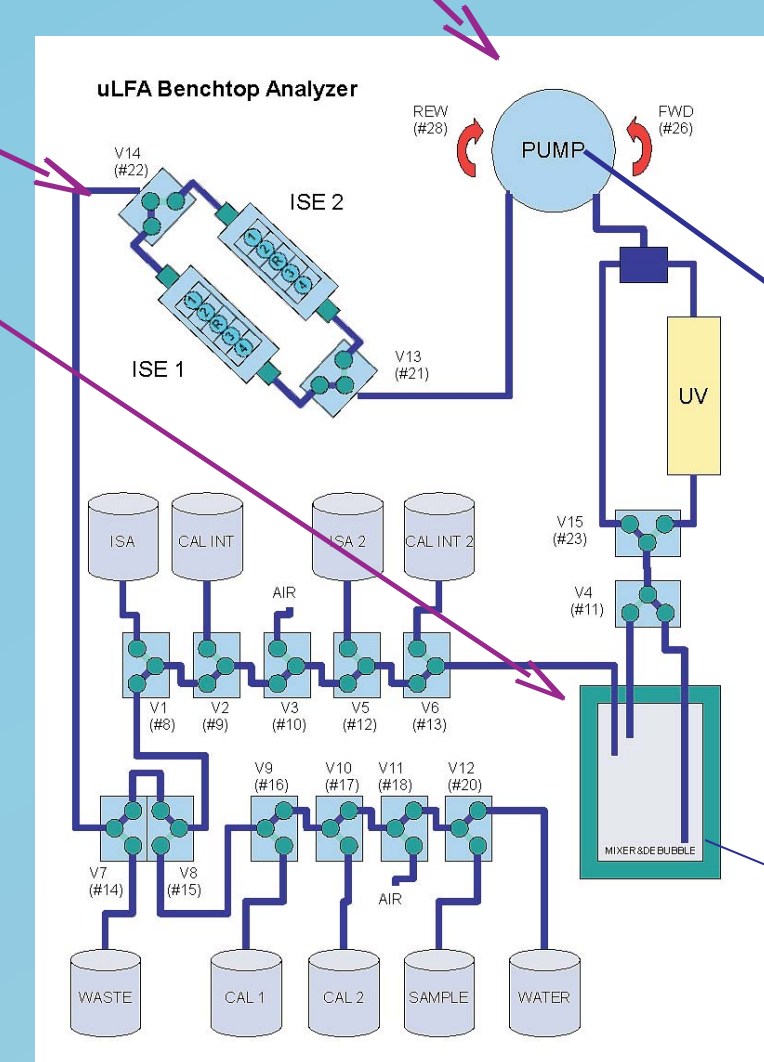
## MicroLoop Flow Analyser: architecture and implementation overview



Laboratory version of MicroLoop Flow Analyser



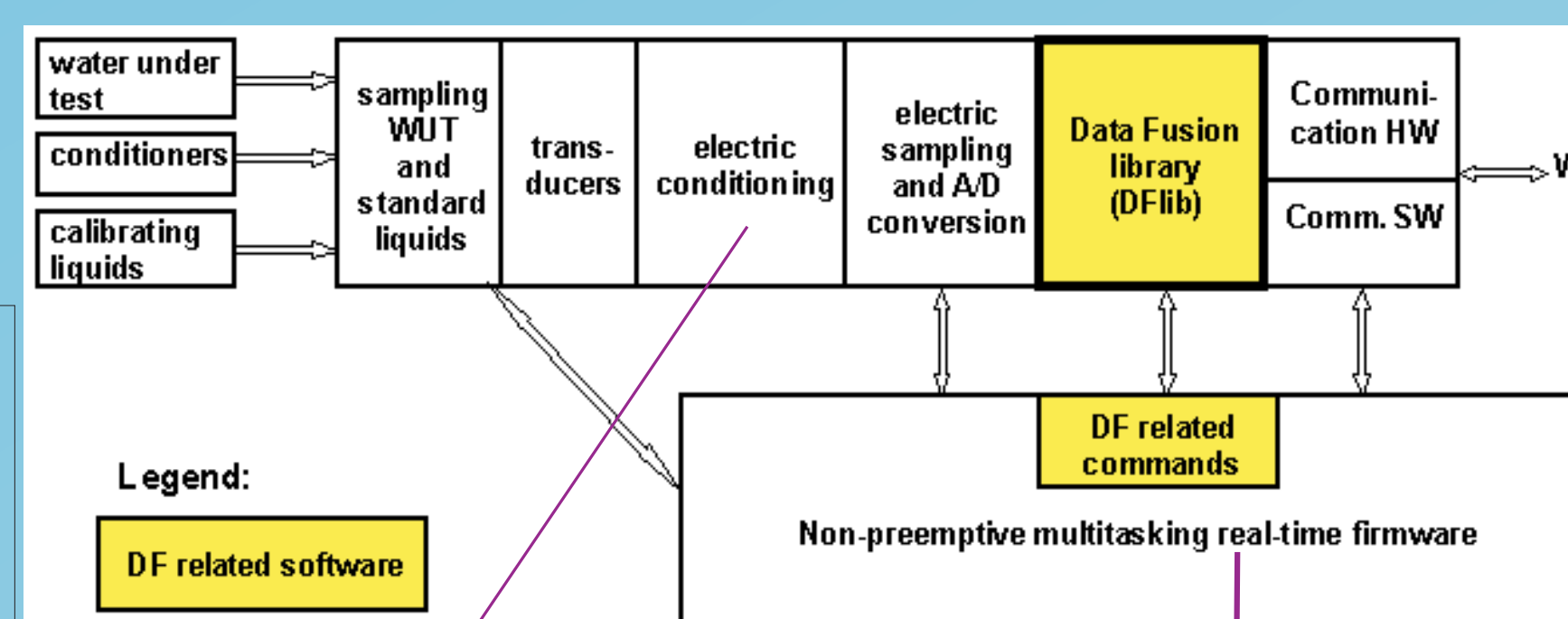
Air bubbles can degrade precision and accuracy of measurements. The optoelectronic refractive bubble detector enables automatic bubble-removal procedures.



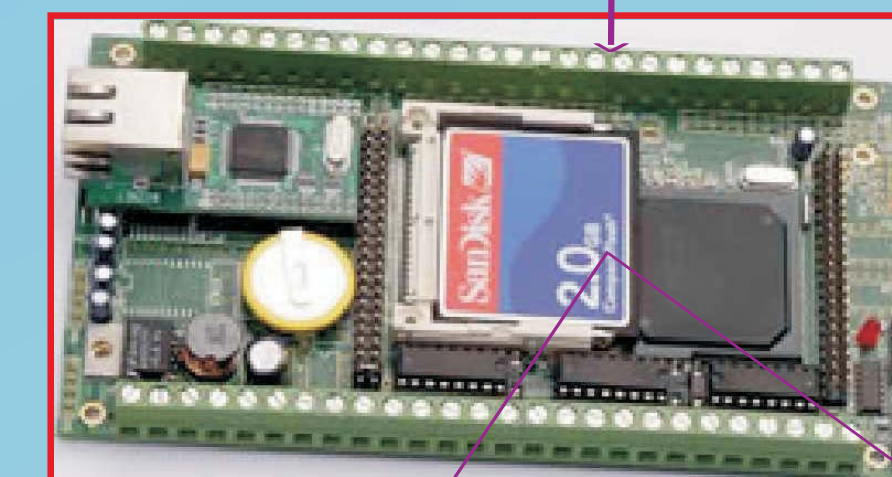
Precise analogue preamplifiers with guard shielding, JFET inputs and 100GΩ input impedance

Bi-directional peristaltic pump enforces flow of liquids, including dosing of standards. It can be run in continuous or PWM mode.

De-bubbling outlet of the mixer device enables efficient air removal from the loop.

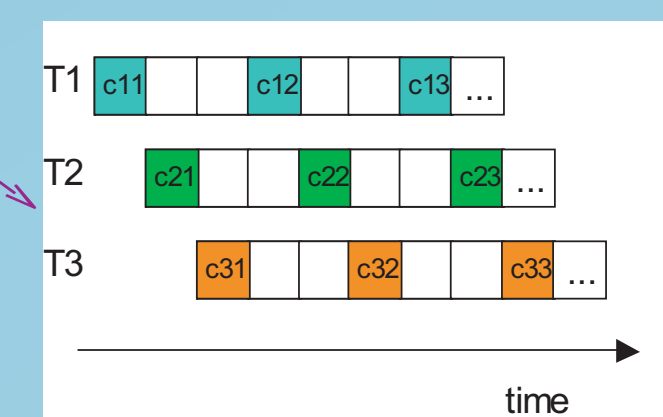


Legend: DF related software



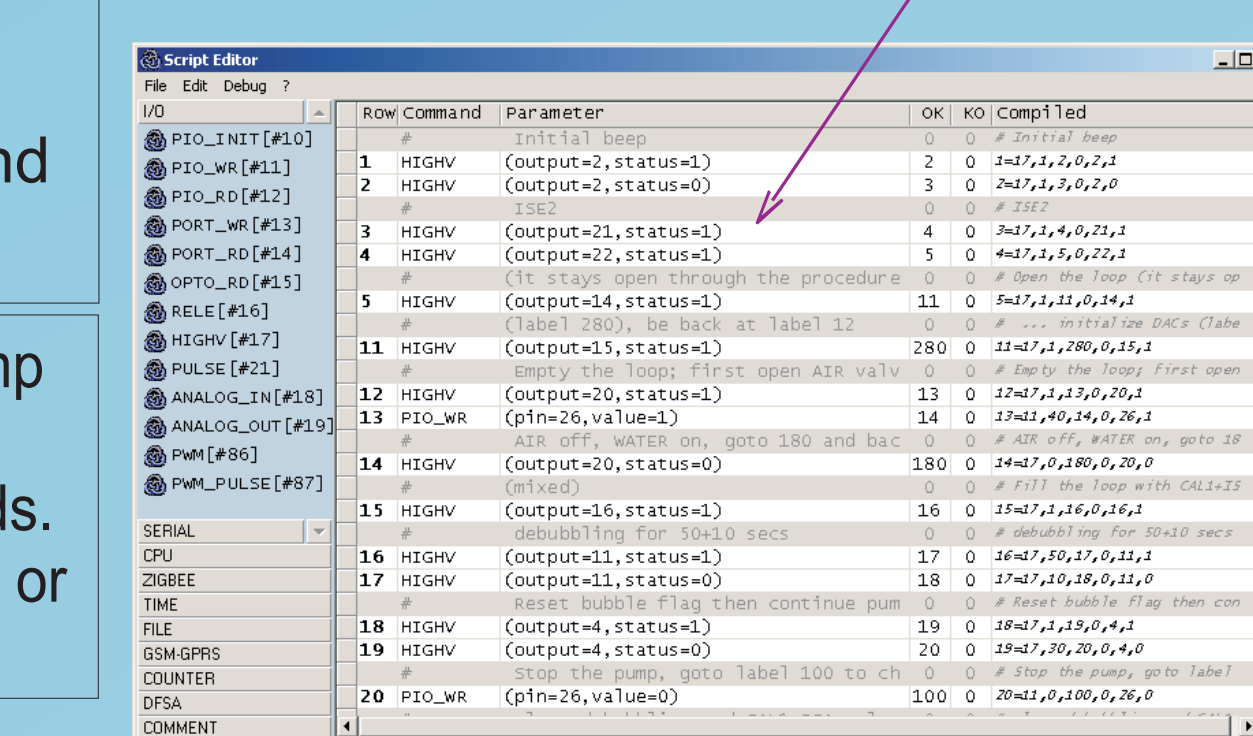
Tern 5D uP board + Systema add-ons enable:

- A/D and D/A
- control of pump and valves
- Compact Flash storage (FAT)
- GSM/Internet connectivity
- 586 processor computational capability



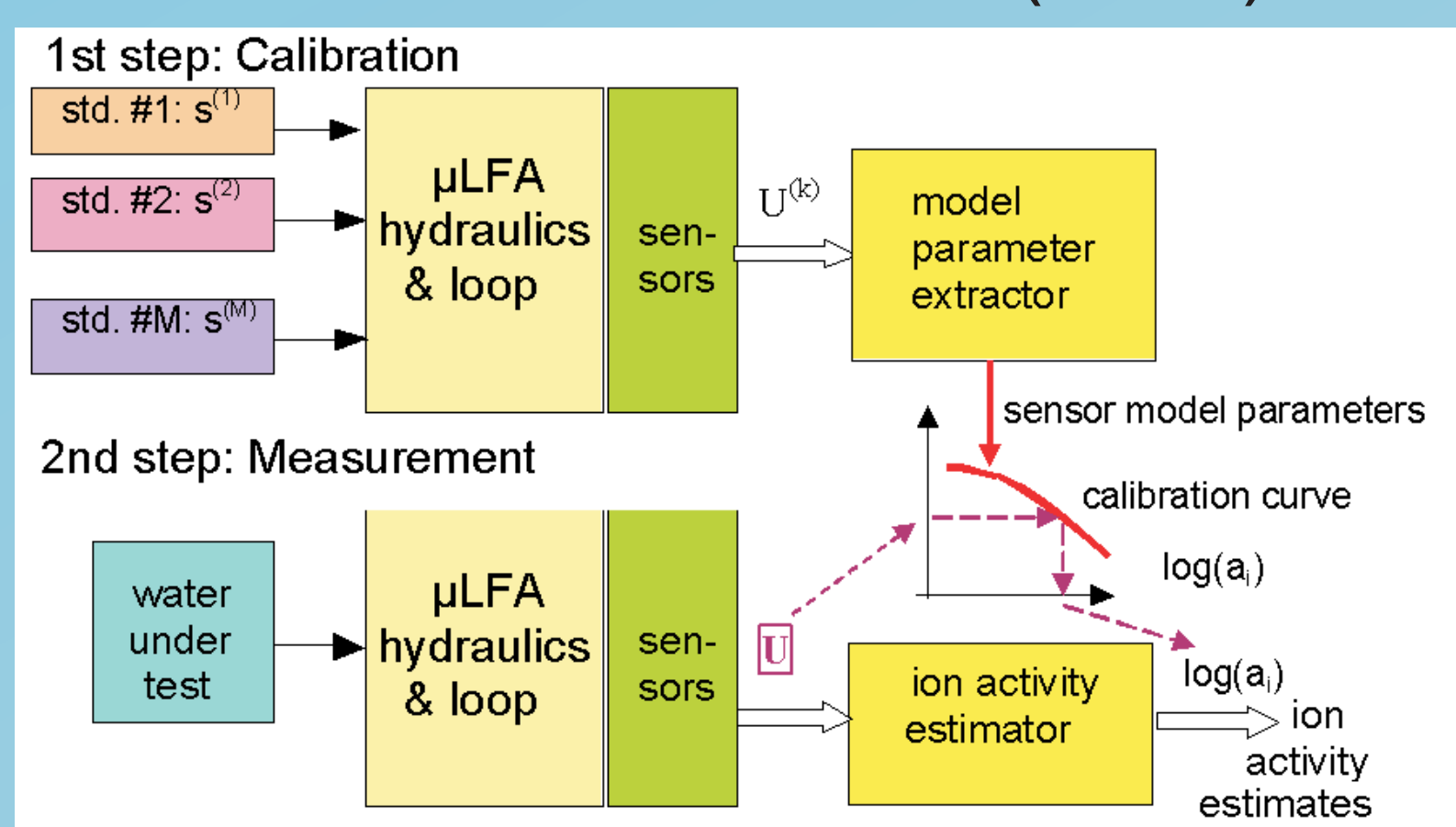
Systema firmware features:

- scripting capability
- non-preemptive Multitasking
- control of uLFA hardware
- remote communication



Measurement cycles are described with a script based language. Integrated Development Environment includes a script editor, file up/downloader and debugger/waveform viewer - for remote uLFA

## Precalibration method (PCM)



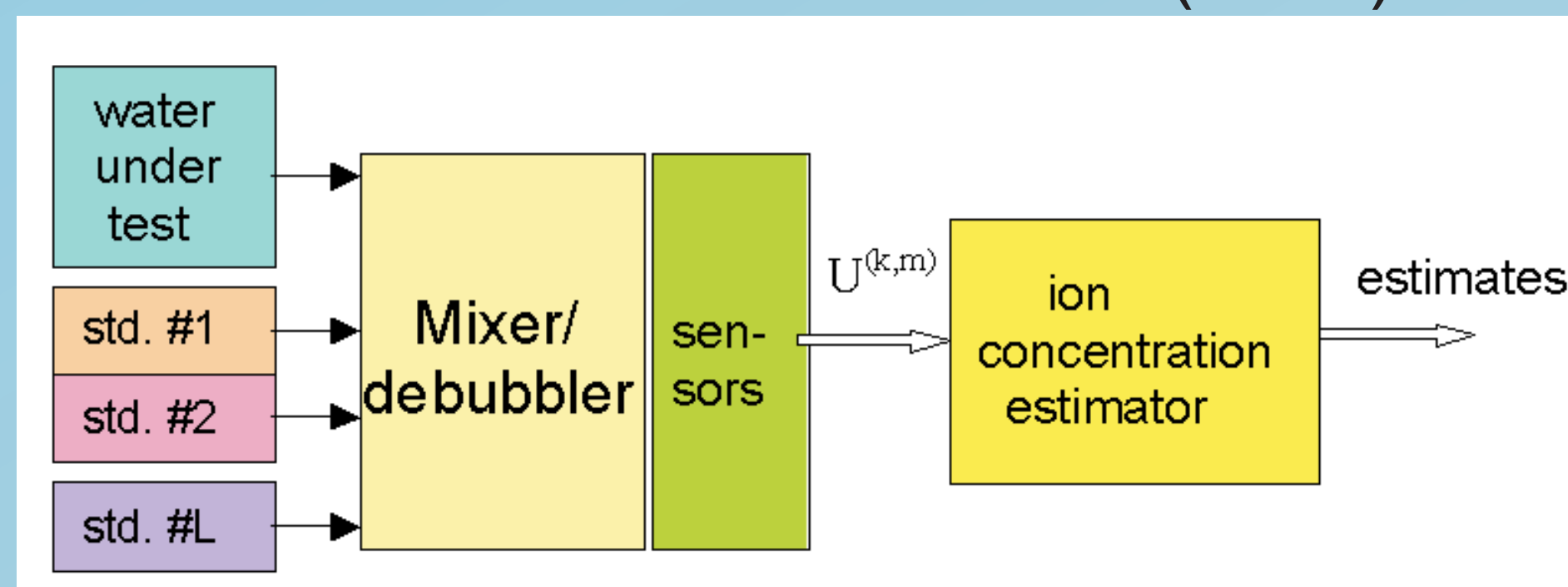
Two measurement techniques have been tested with the uLFA laboratory unit:

- Pre-Calibration based Method (or PCM) - performs sensor calibration prior to interpreting read-out for the sample under test
- Standard Addition Method - takes read-outs for the sample, and after each precise addition of standard liquids is made

Note: scripting capability of uLFA makes addition of other measurement procedures possible.

## Measurement techniques

### Standard addition method (SAM)



### Multiparameter measurement

- Consequence of limited selectivity of IES:
  - response depends also on interfering ions, perhaps of no direct interest
  - limit of detection depends on concentration of interfering ions.
- Data Fusion concept: process read-outs of several sensors together, to compensate for interferences and improve accuracy.
- ULFA integrates a WUT-developed library of numerical algorithms implementing the DF concept. Thus multiparameter ion estimation is accessible in measurement scripts.

### SAM PCM

- ✓ (Small) volume of liquids used
- ✓ Resistance to ion matrix effect
- ✗ Precision

## Example multiparameter measurement

### Setup:

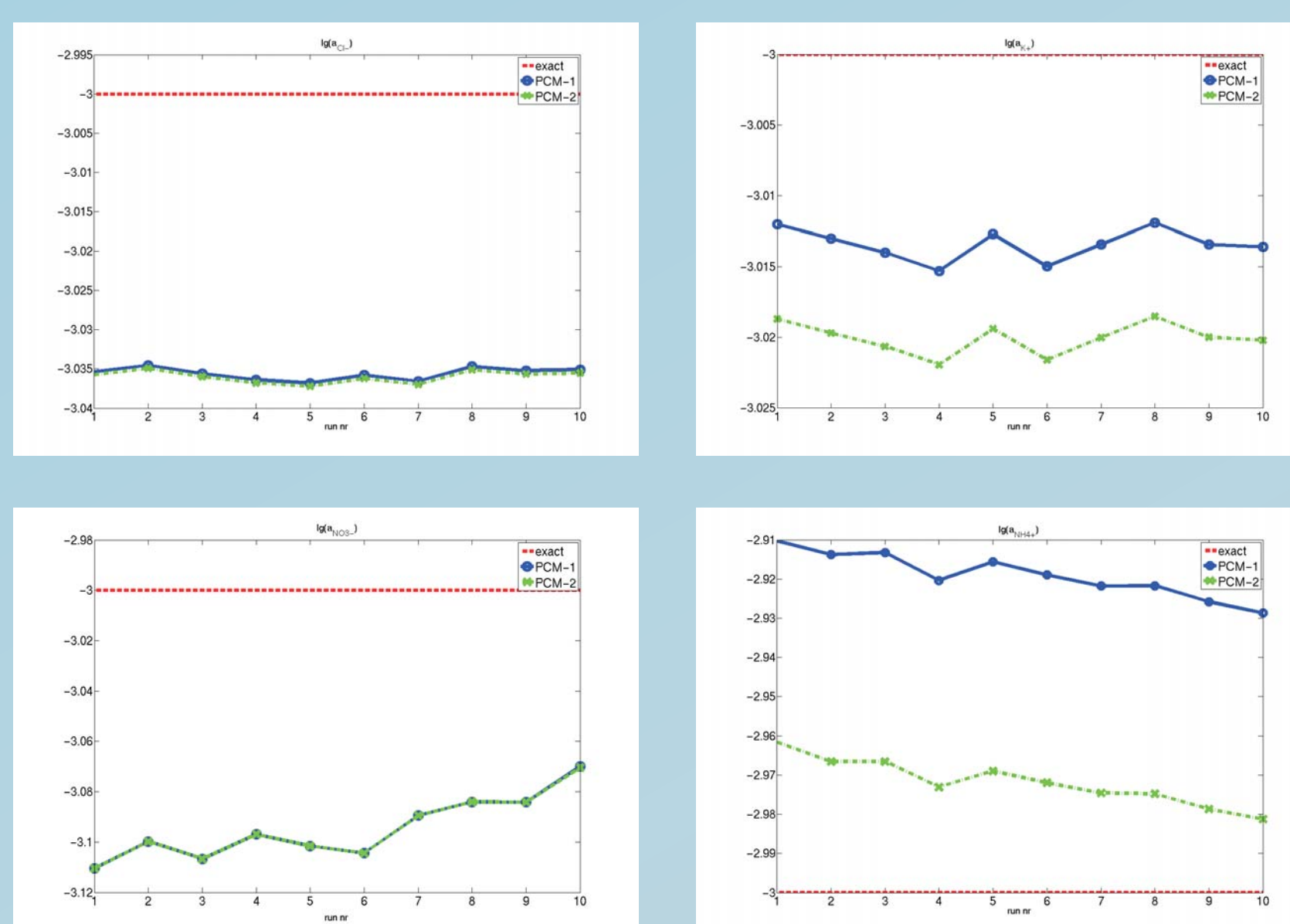
- sample contains 1mM of  $\text{NH}_4\text{NO}_3$  and 1mM of KCl
- 4 miniaturised sensors sensitive to  $\text{NO}_3^-$ ,  $\text{NH}_4^+$ ,  $\text{Cl}^-$ ,  $\text{K}^+$  ions
- 10 repetitions of measurement procedure

$\text{NO}_3^-$  and  $\text{NH}_4^+$ : ions of main interest  
 $\text{Cl}^-$ ,  $\text{K}^+$ : interfering ions

### Results:

- $\text{NH}_4^+$ : significant improvement of accuracy, no degradation of precision.
- $\text{NO}_3^-$ : sensor works at its linear range => no real improvement of accuracy possible
- $\text{K}^+$ : slight deterioration of accuracy (interfering ion)

## Capability of MicroLoop Flow Analyser (uLFA) with miniaturised $\text{NO}_3^-$ , $\text{NH}_4^+$ , $\text{Cl}^-$ , $\text{K}^+$ sensitive sensors



PCM-1 - one-sensor-at-a-time measurement  
 PCM-2 - multiparameter (Data Fusion based) measurement

### Major features of uLFA

- script based programmability of measurement cycles
- good repeatability of measurements
- multiparameter analysis capability (up to 8 ions)
- ready for autonomous long-term operation
- remote access and control enabled