LRTAP Long-range Transboundary Air Pollution

2nd Meeting of the Heads of the Laboratories, 12.-13. October 2009, Warsaw, Poland

Measuring of elements in aqua regia digestion solutions with ICP – problems and solutions

Nils König and Karl-Ludwig Lüter, Göttingen, Germany

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problems:

- **aqua regia matrix (Cl-compounds)**
- **soil matrix (high Al, Fe, sometimes Ca, low heavy metal conc.)**
- **calibration standard matrix (standards with low or high conc. for all elements!)**
- **wavelength interferences between elements**

solutions ???

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our ICP instruments:

Thermo Scientific

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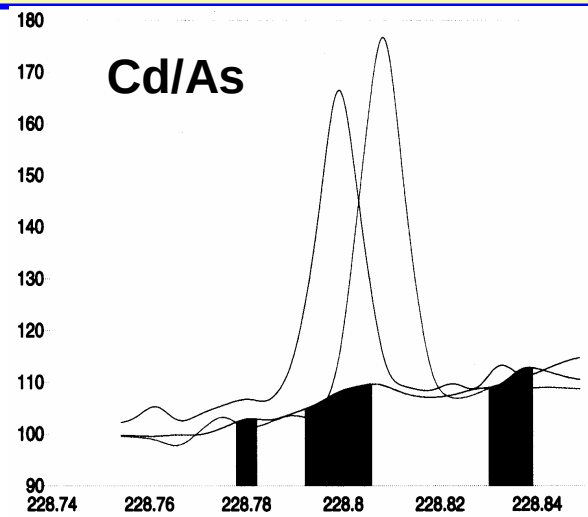
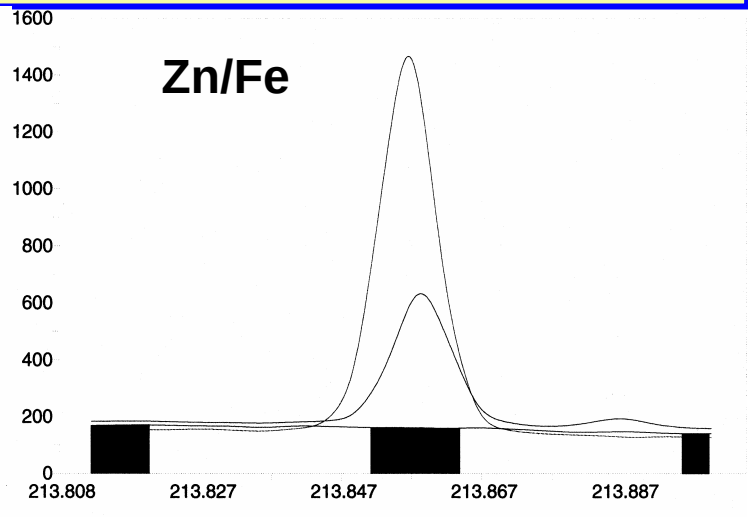
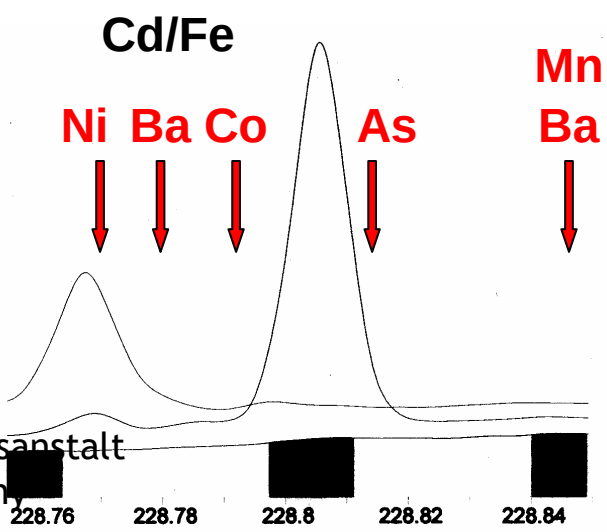
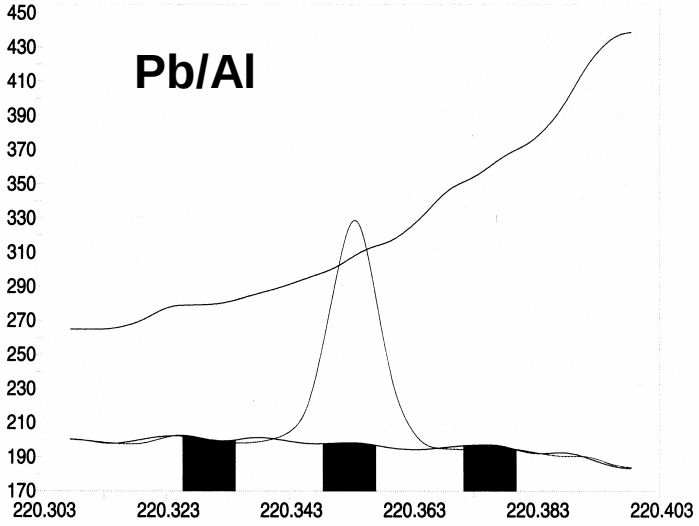
wavelength interferences between elements (examples):









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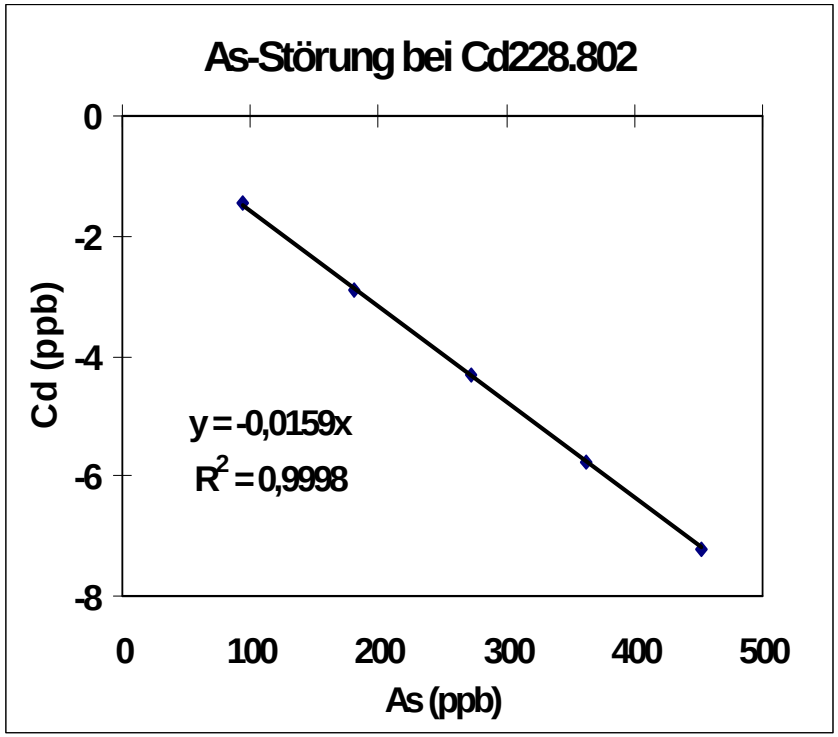
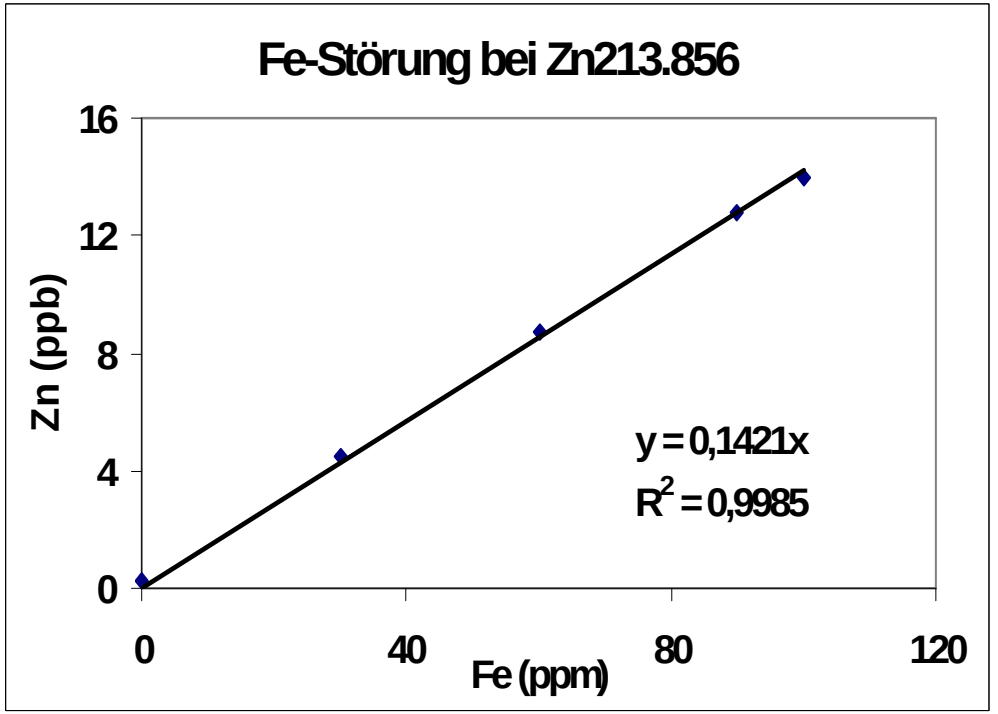
Solution: Interelement correction





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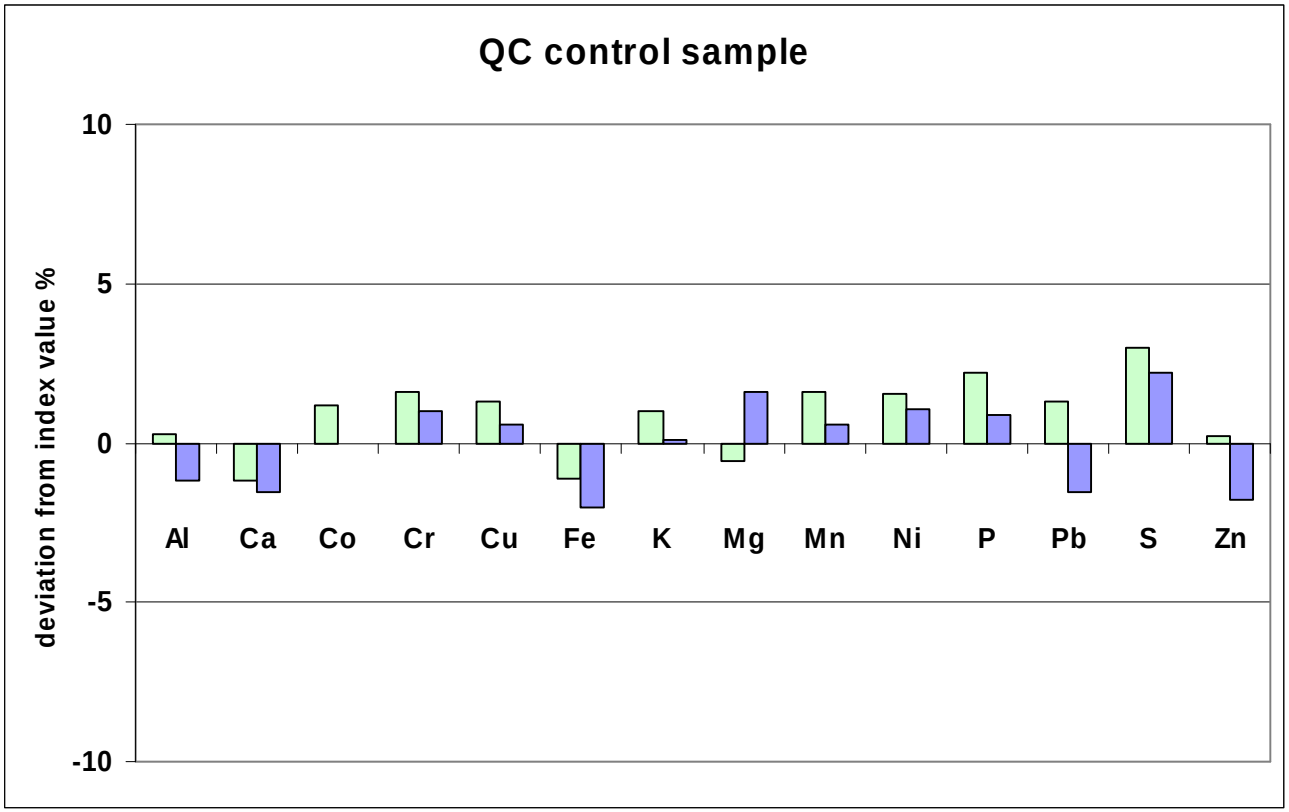
Influence of the aqua regia matrix compared to HNO_3 matrix:



HNO_3 matrix

aqua regia matrix

=> no difference, normal variation of 1-3 %

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effects of the soil matrix (high Al, Fe, sometimes Ca, low heavy metal conc.) to the ICP plasma:

High salt conc. => cooling of the plasma => worse atomisation for some elements, lower ionisation of alkaline elements

effect: apparently higher Na, K conc. and lower P conc.

2 possible solutions:
- adaption of the calibration standards
- internal standards

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Influence of adapted and non-adapted calibration standards:

Comparison of results after 2-point calib. (zero and high std.) and adapted 4 point calib. (4 std. with similar salt conc.)

2-point-cal.	High cal. standard
Element	Konz
Cd	200 ppb
Co	200 ppb
Cr	200 ppb
Cu	200 ppb
Ni	200 ppb
Pb	2000 ppb
Zn	2000 ppb
Al	100 ppm
Ca	100 ppm
Fe	100 ppm
K	40 ppm
Mg	40 ppm
Mn	20 ppm
Na	20 ppm
P	20 ppm
S	20 ppm
Ti	8 ppm

4-point-cal	Std 1	Std 2	Std 3	Std 4
Element	Konz	Konz	Konz	Konz
Cd	50 ppb	100 ppb	150 ppb	
Co	100 ppb	200 ppb		
Cr	100 ppb	200 ppb	300 ppb	
Cu	100 ppb	200 ppb	300 ppb	
Ni	100 ppb	200 ppb		
Pb		2000 ppb		
Zn	400 ppb	800 ppb	1200 ppb	
Al	2 ppm	20 ppm	40 ppm	100 ppm
Ca	20 ppm	40 ppm	1 ppm	100 ppm
Fe	2 ppm	20 ppm	100 ppm	40 ppm
K	5 ppm	1 ppm	40 ppm	20 ppm
Mg	10 ppm	2 ppm	40 ppm	20 ppm
Mn	2 ppm	10 ppm	20 ppm	
Na	2 ppm	10 ppm	20 ppm	
P	2 ppm	10 ppm	20 ppl	
S	2 ppm		20 ppm	
Ti			8 ppm	2 ppm

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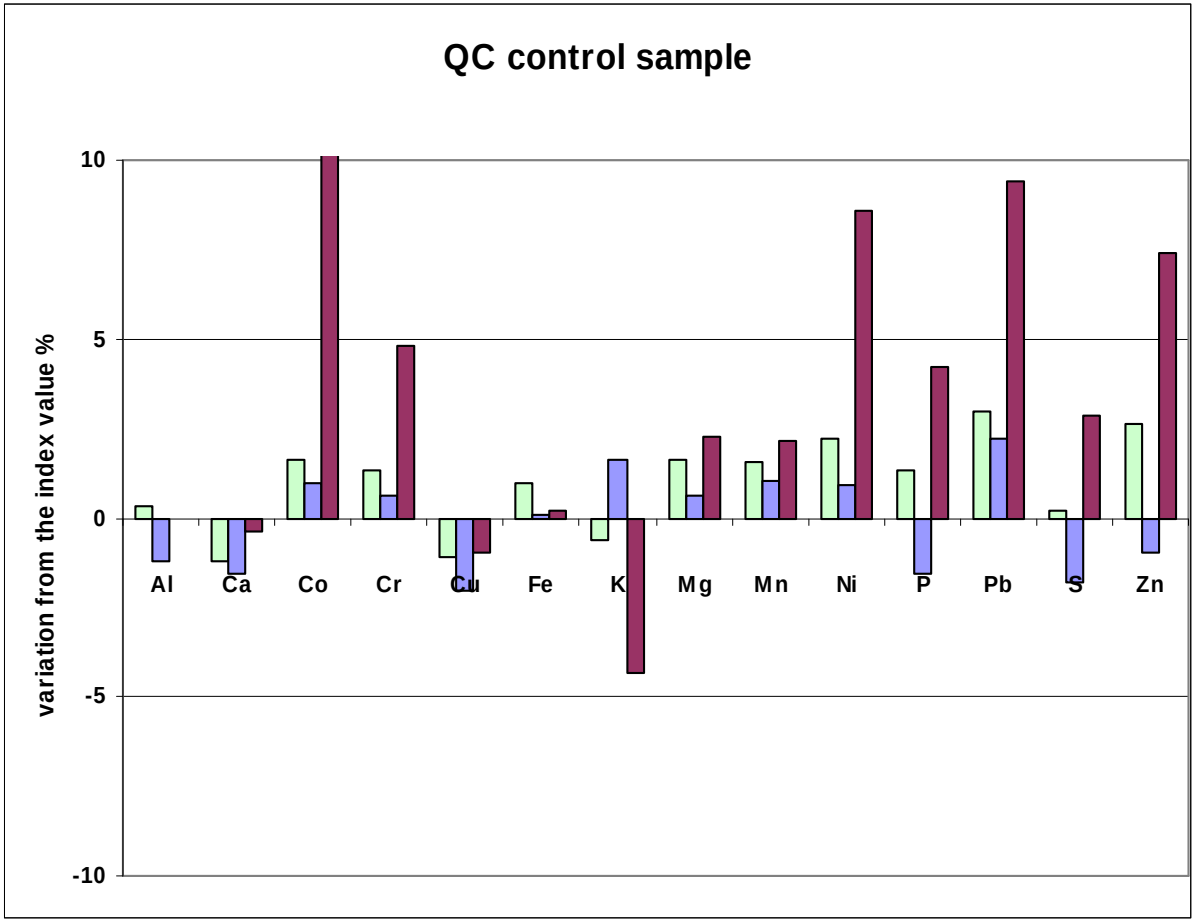
adapted 4 point calib. HNO_3 matrix

adapted 4-point calib. aqua regia matrix

2 point calib. (zero/high)

=> For some elements variations from – 5 % (K) to + 5 (P) or + 10 % (heavy metals)



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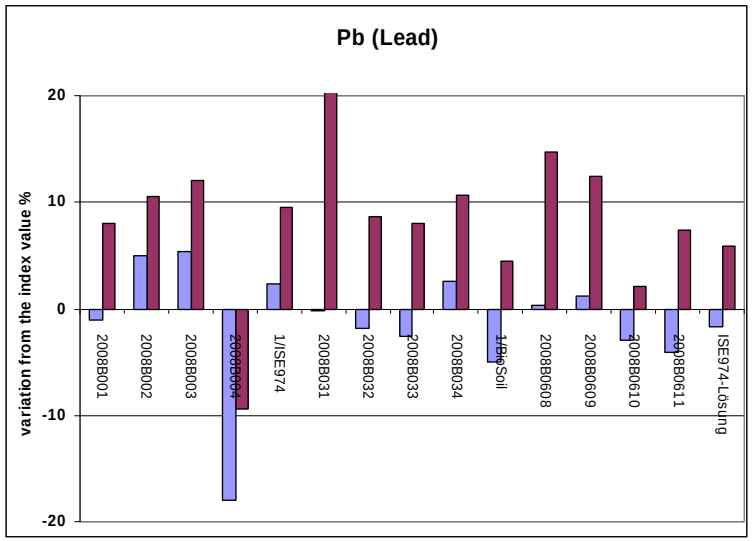

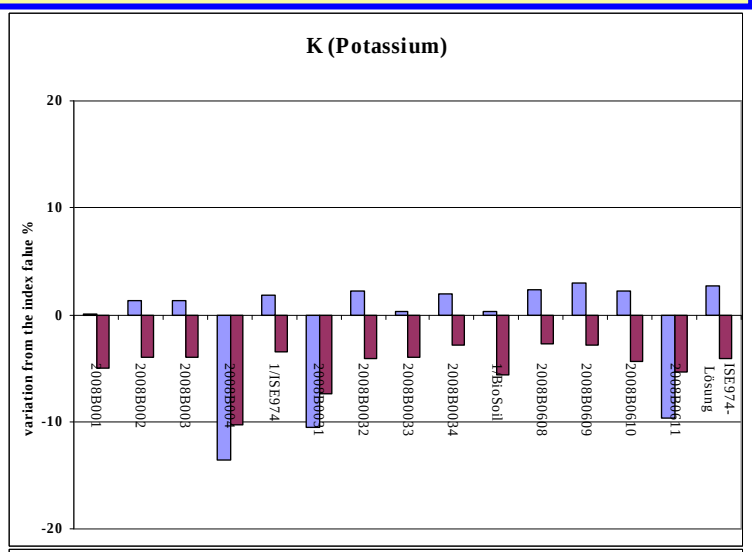

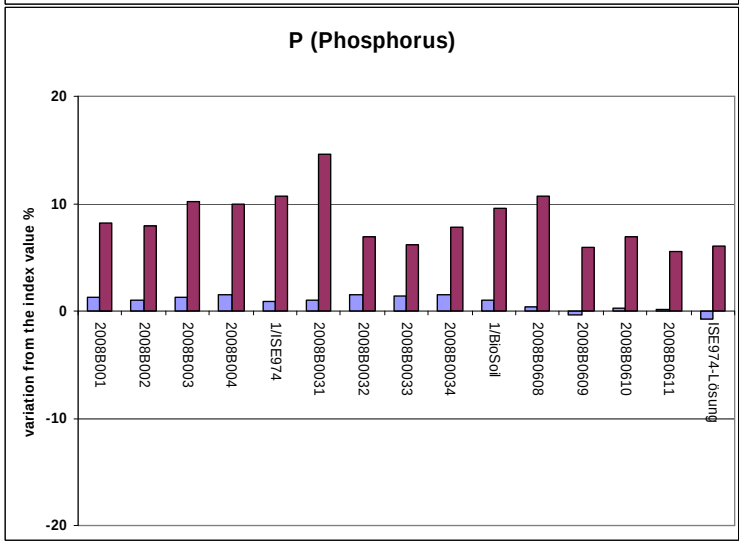

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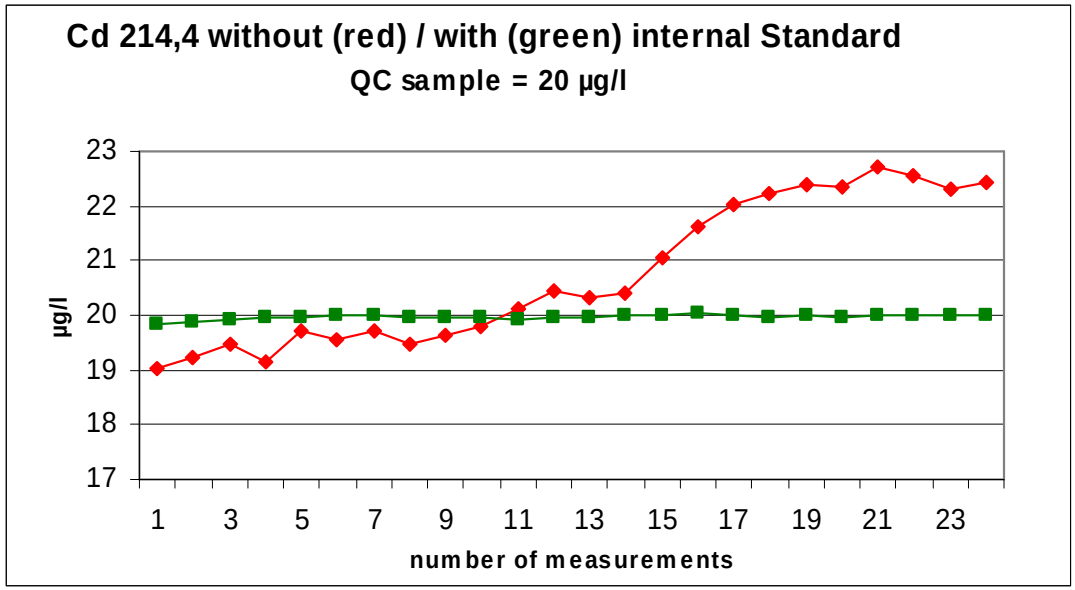
Use of internal standard:

Good internal standards react on changings of the viscosity, the salt concentration and the plasma temperature the same way as the corrected elements in the plasma:



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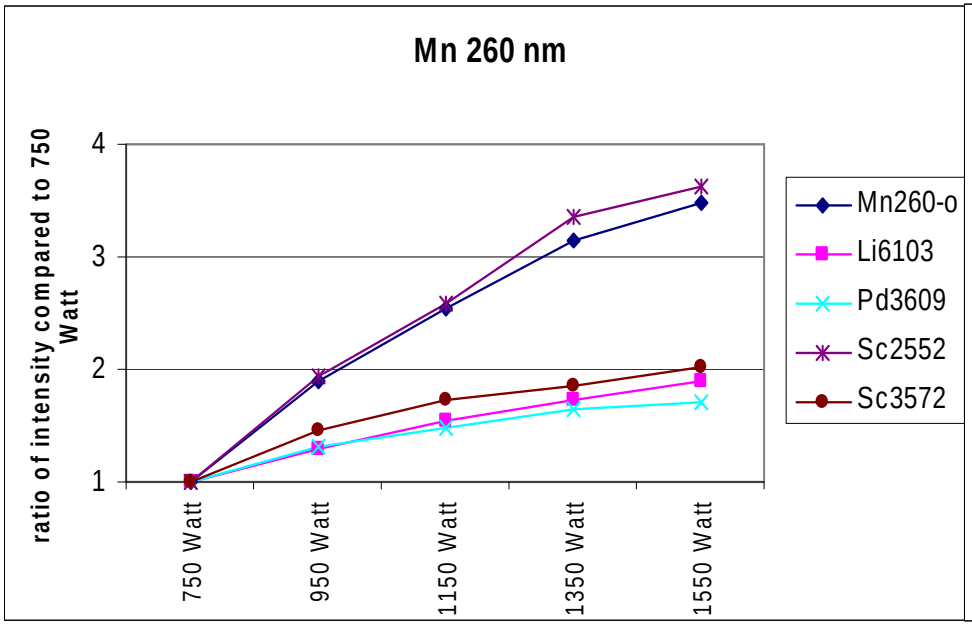
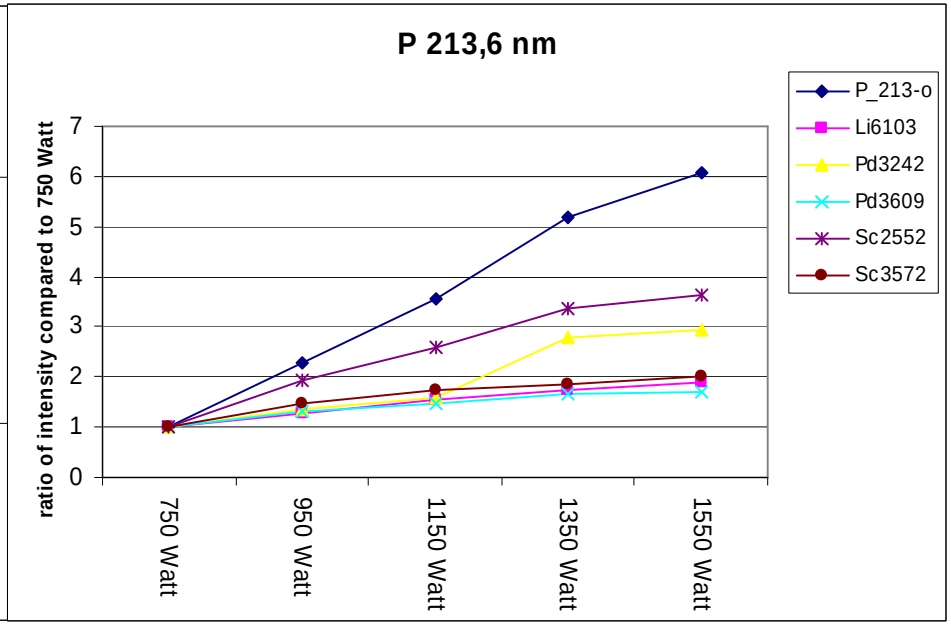
Use of internal standard:

The problem is that different elements lines react sometimes different from the internal standard lines:





=> For each used element line a adapted internal standard is needed!

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Thank you

for

your interest !