



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

**open aqua regia, HNO_3 -pressure
and HNO_3 -microwave digestion
for humus samples:
comparison
of the coefficients of variation
in a special ring test**

Nils König, Göttingen



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With normal ringtests it is difficult to find the reasons for bad ringtest results specially for analytical methods with 2 or more steps during the analysis

1. step: extraction/digestion
2. step: measurement of elements in the extract/digestion solution

Which step is the reason for high variations of the results ?

**To clarify this problem
a new ringtest method was developed:**



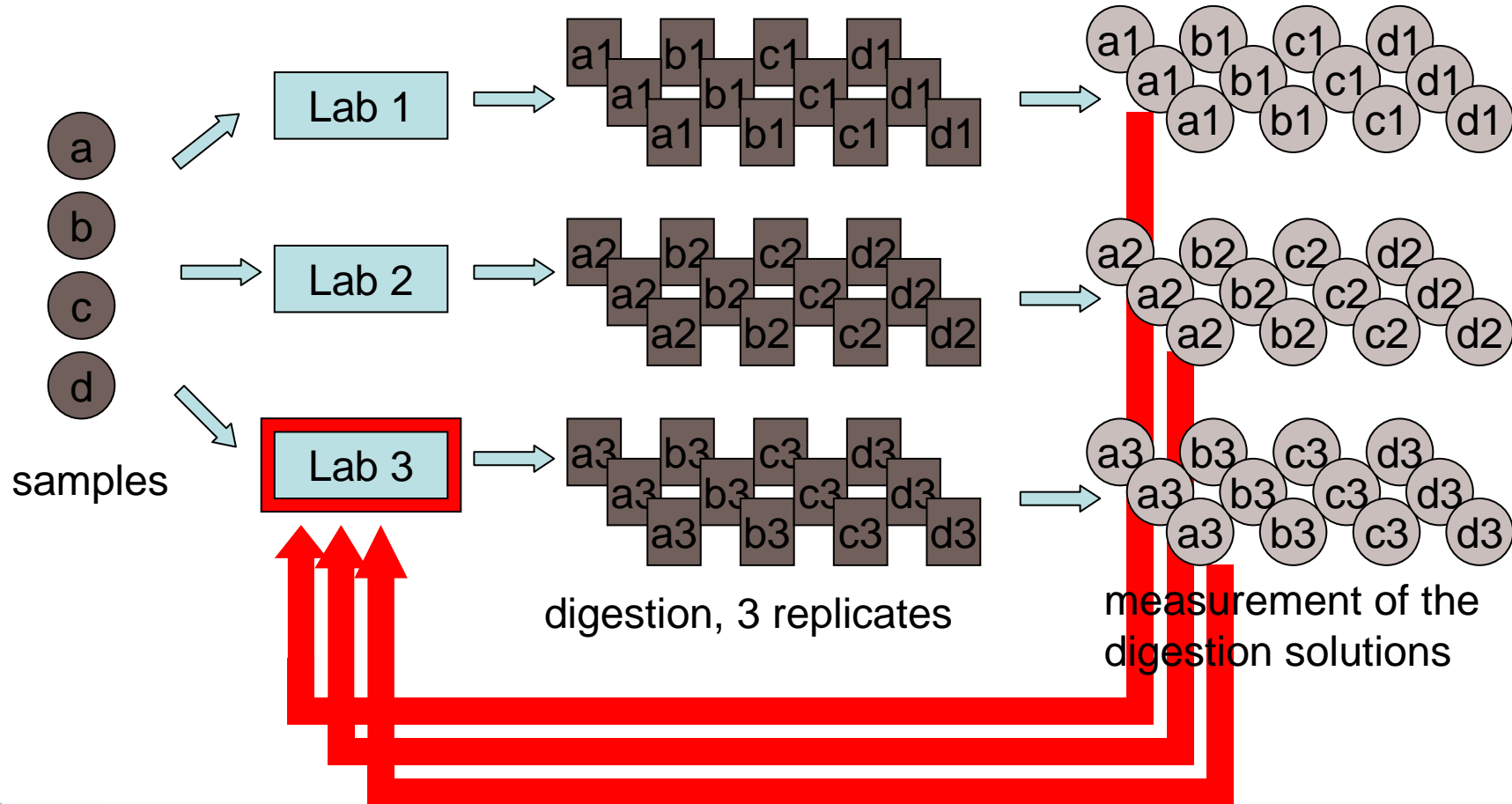
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digestion method	number of labs	number of samples (initial measurement)	number of samples (control measurement)
aqua regia digestion	11	7	3
HNO₃ pressure digestion	8	7	3
HNO₃ microwave digestion	4	7	3



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HNO₃ pressure digestion

200 mg of a humus sample are weighed in a pressure digestion vessel of one's choice and mixed with 4 ml of concentrated HNO₃ (highly pure). Digestion will take at least 6 hours at a minimum temperature of at least 170°C. After cooling off, digestion residue is filtered out through a highly sensitive filter ("Schwarzband" filter) or membrane filter washed with acid into a 50 ml volumetric flask. Rinsing of the digestion vessel and the filter, as well as filling up of the digestion solution to 50 ml, are done with 0.65% HNO₃.

HNO₃ microwave digestion:

200 mg of humus sample are weighed into the digestion vessel and mixed with 4 ml of concentrated HNO₃ (highly pure). **Digestion conditions are to be chosen depending on the equipment.** After the digestion vessels have cooled off, the digestion residue is filtered out through a highly sensitive filter ("Schwarzband" filter) or membrane filter washed with acid into a 50 ml volumetric flask. Rinsing of the digestion vessel and the filter, as well as filling up of the digestion solution to 50 ml, are done with 0.65% HNO₃.



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Aqua regia digestion

1 g of humus sample is refluxed for 2 hrs with 20 ml aqua regia (15 ml concentrated HCl p.a. and 5 ml concentrated HNO₃ p.a.). After cooling off, digestion residue is filtered through a highly sensitive filter ("Schwarzband" filter) or membrane filter rinsed with acid into a 100 ml volumetric flask. Rinsing of the digestion glass and of the filter as well as filling up of the digestion solution to 100 ml take place with 0.65% HNO₃.



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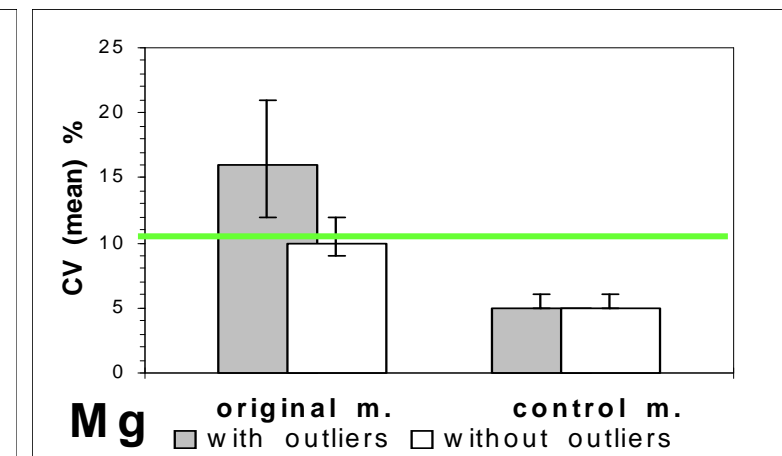
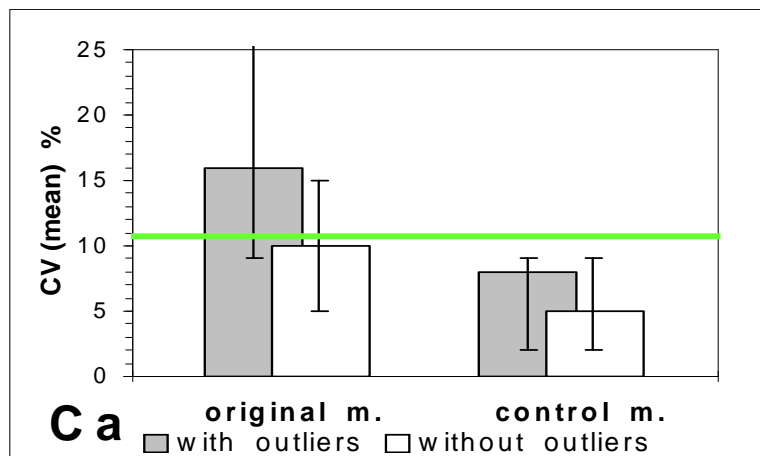
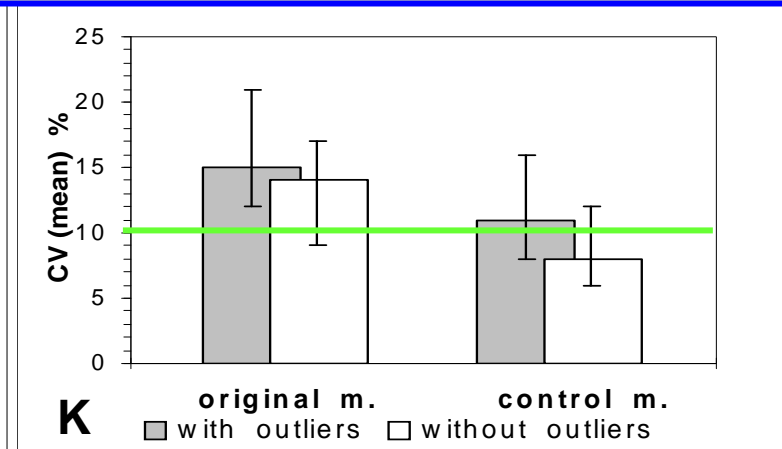
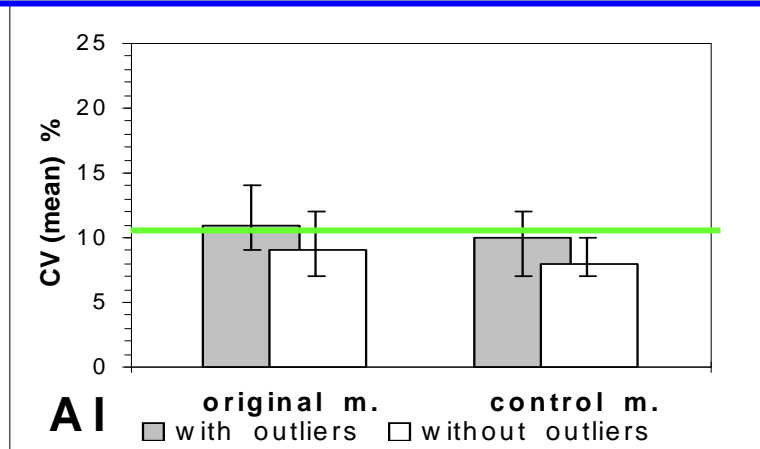




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open
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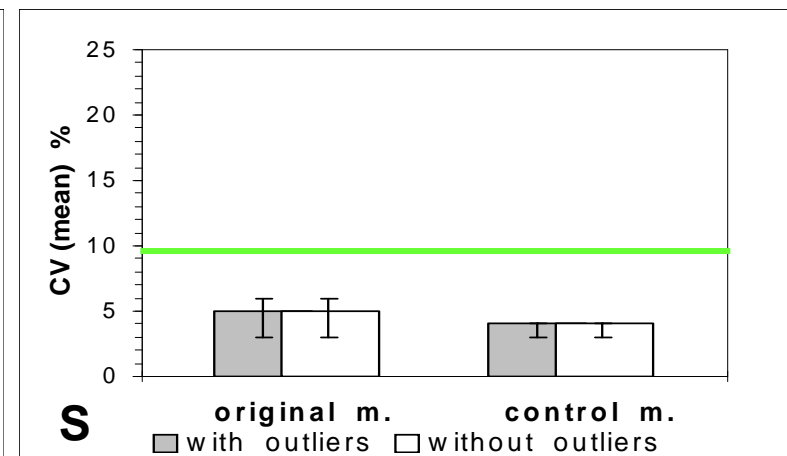
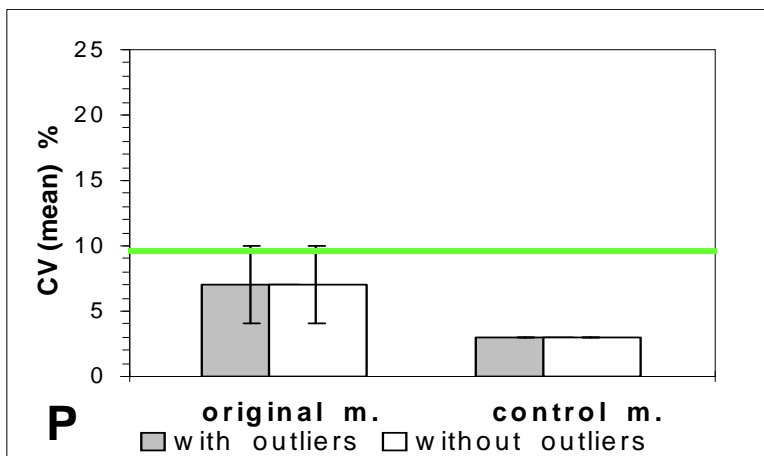
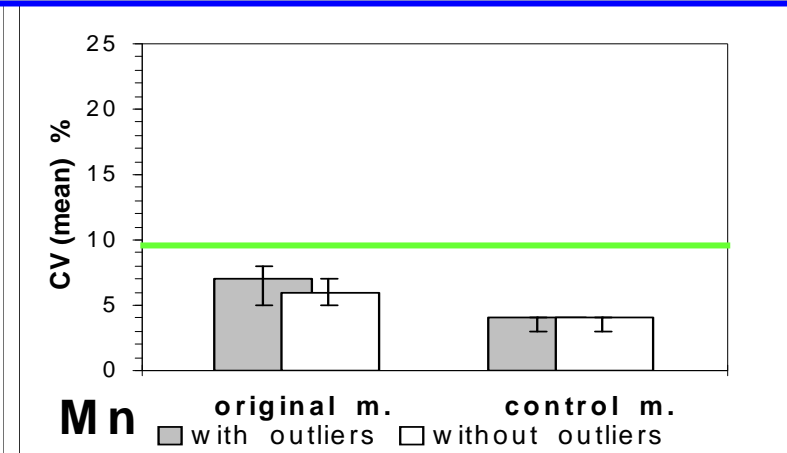
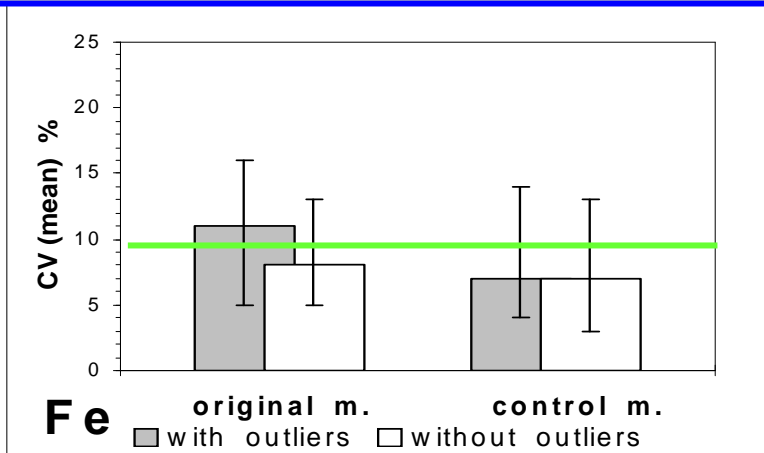
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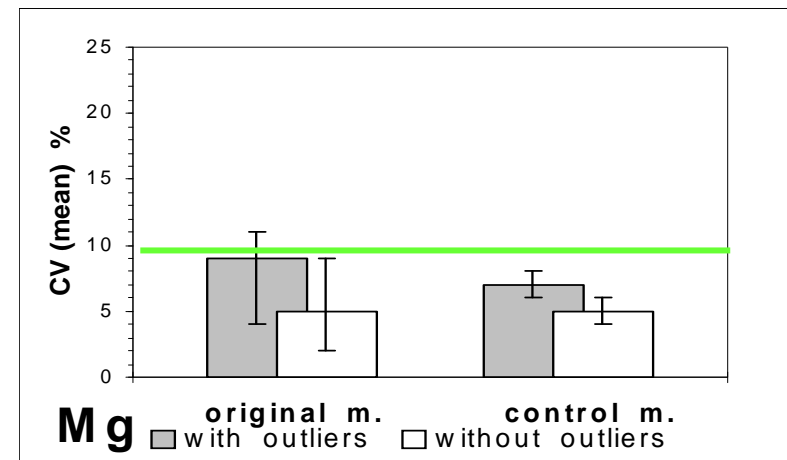
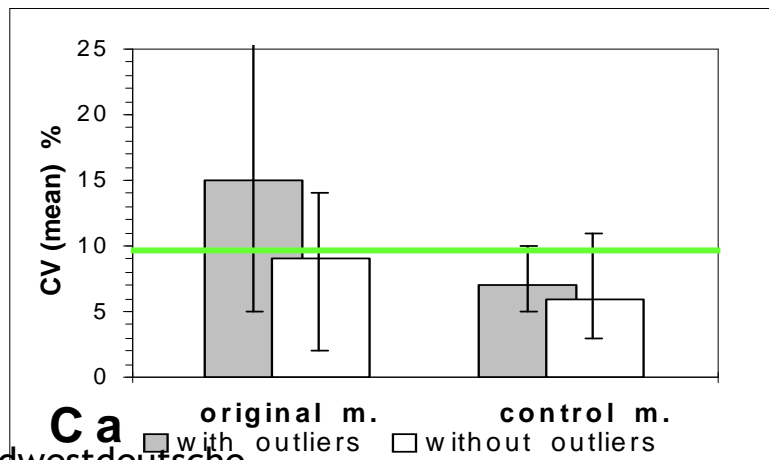
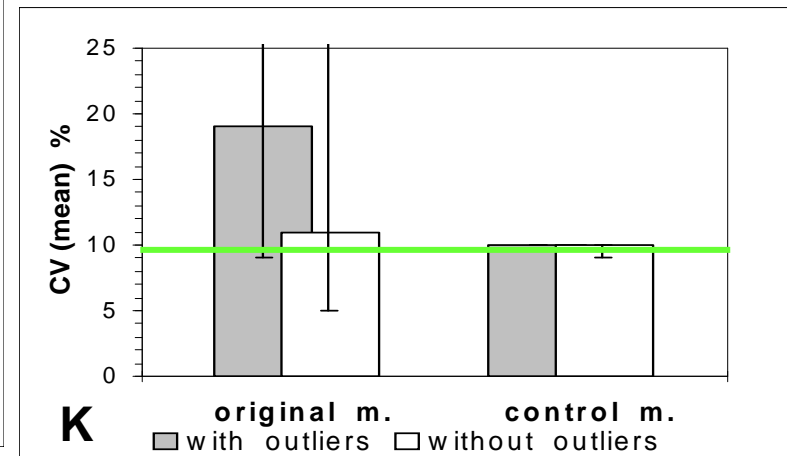
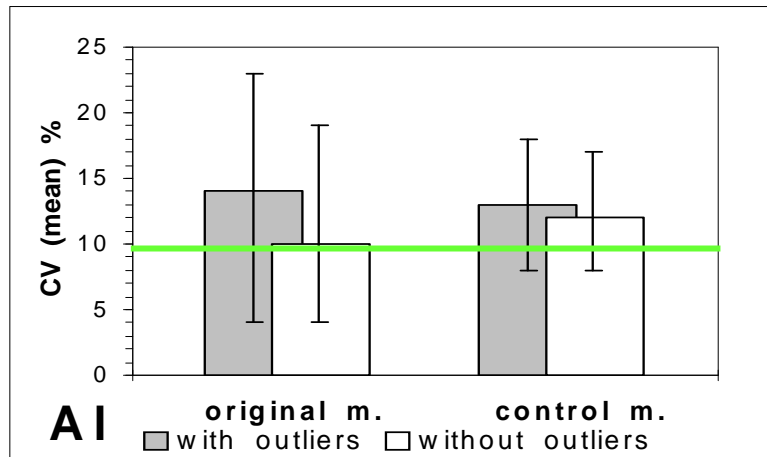
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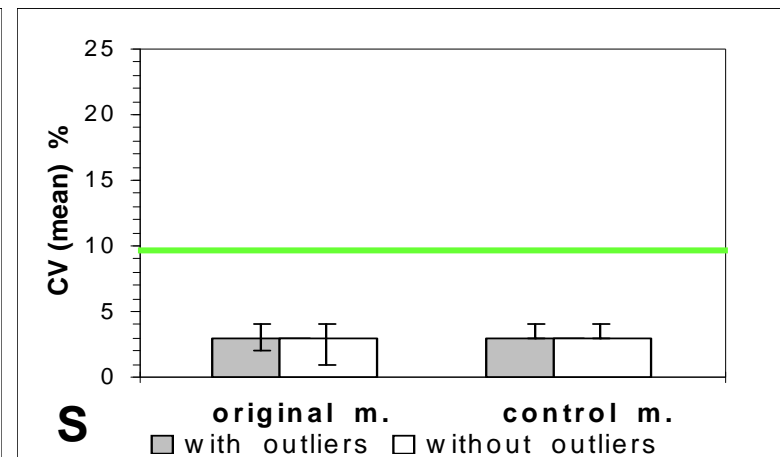
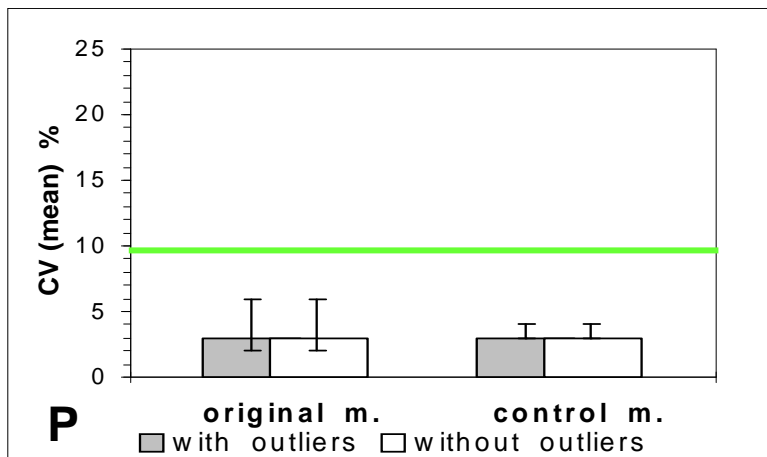
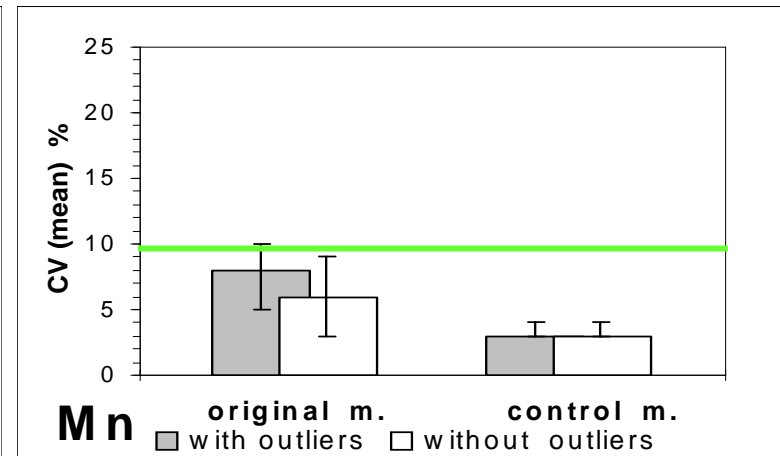
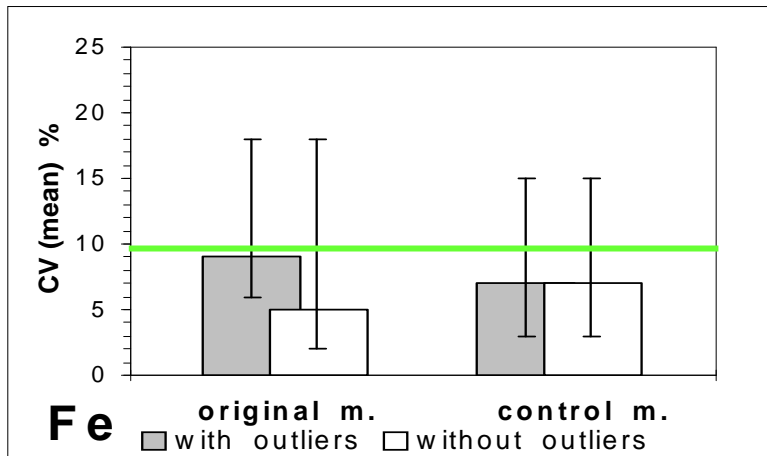
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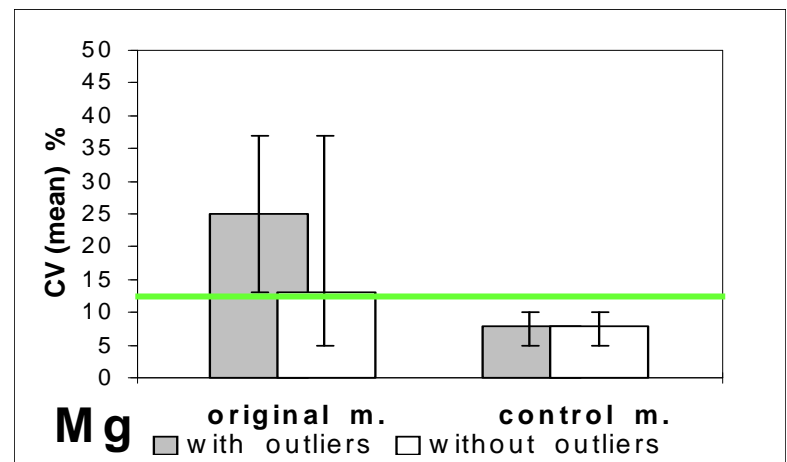
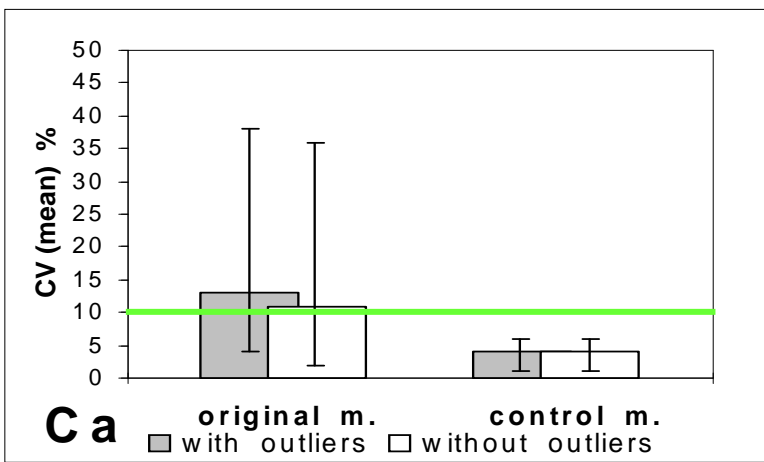
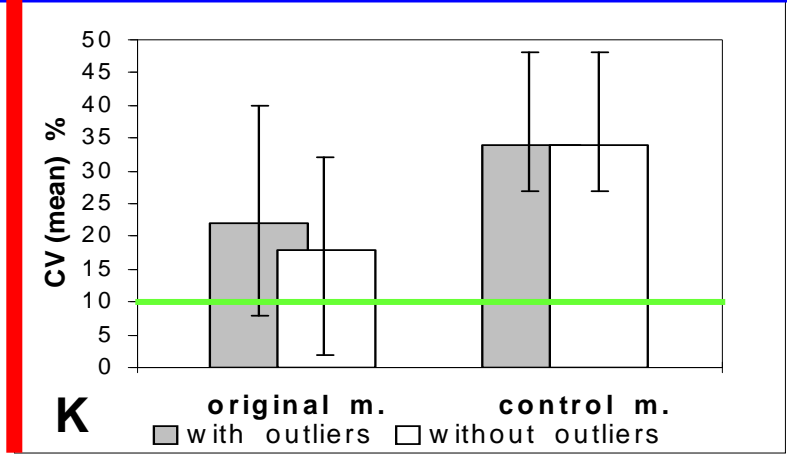
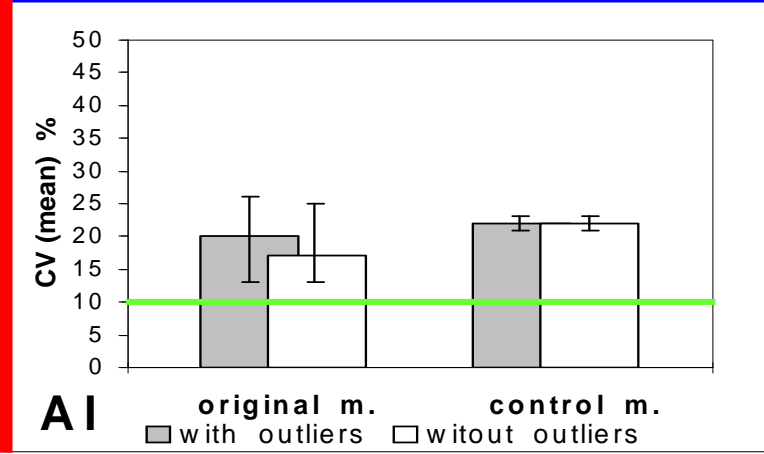




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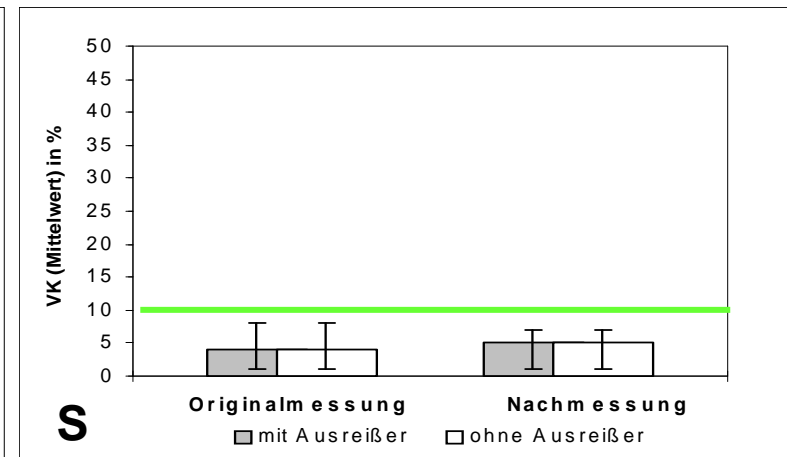
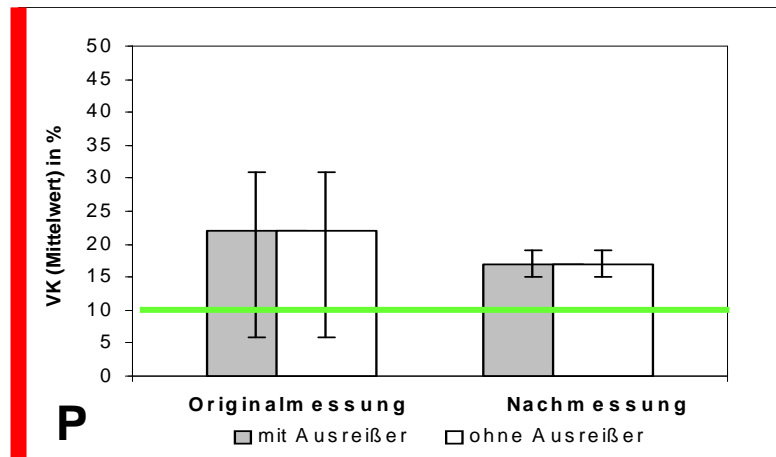
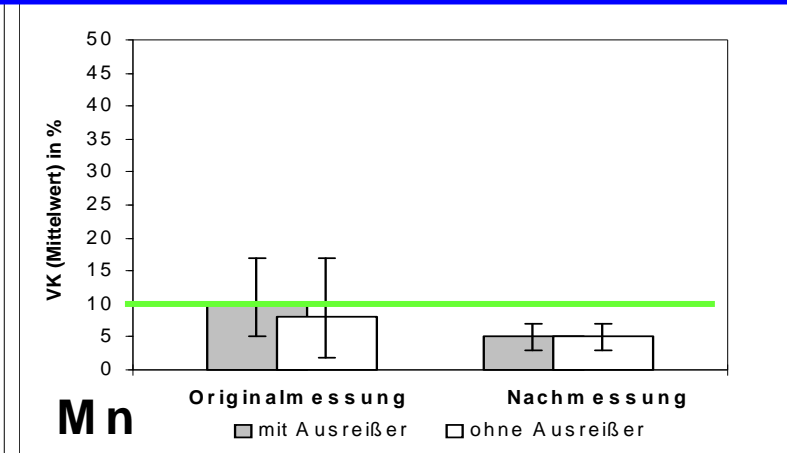
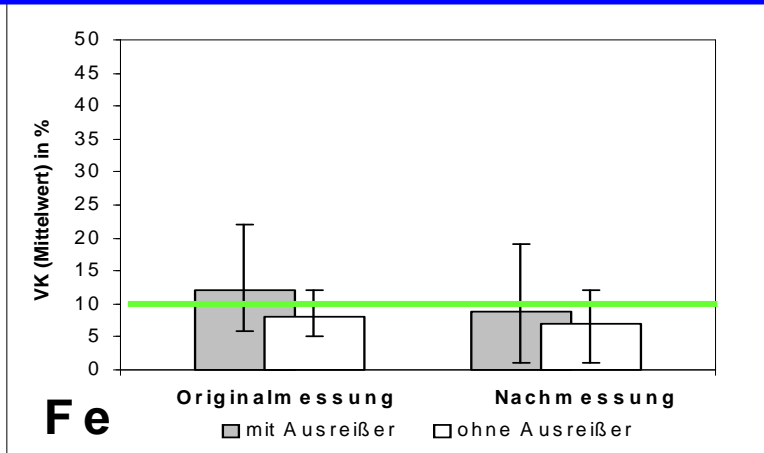


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Conclusions:

- For the elements Al, K and P the coefficients of variation (CV) are much higher for the HNO₃-microwave digestion than for the HNO₃-pressure digestion or the aqua regia digestion

- the reason is that it is very difficult to define the conditions for the energy supply (strength, time) in different microwave systems; for matrices which are not digested completely the different conditions lead to different digestion results

- in humus samples the mineral phase cannot be digested completely with HNO₃ or aqua regia in a microwave system; therefore the completeness of the digestion is a function of energy and time

=> Microwave digestion is not usable for the aqua regia digestion of humus or soil samples or the HNO₃ digestion of plant material for the element Al and sometimes K and P



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**Thank you
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