Accidental contamination of water samples in the laboratory (for sulphate, chloride, Ca, Mg)

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Concentrations of elements and ions in precipitation samples, specially in bulk deposition, are low and very low. That’s why the possibility of sample contamination in the field and in lab is very high.

Because of the financial situation and varied solicitations, we must perform soil, needles/leaves and precipitation analysis in the same lab.

a) Example of sulphate contamination

- The problem that we’ve met was that one day we obtained a very unsatisfactory calibration curve and no reproducibility of results.
- Usually, the calibration curve was fine (reproducible equation slope, correlation coefficient greater than 0,999, linear equation through zero) and the results were reproducible.
- What happened? Why were the results bad?

- In the same day when we had the problems with sulphate determination, we used our Buchi apparatus for nitrogen determination by Kjeldahl method.
- It seams that the digestion system was not tight fitting enough and the SO3 vapors escaped were fixed by our precipitation samples and enriched them in SO4 ions.
- After that, we took all the measures to avoid the contamination and the problem was solved.

b) Measures to avoid chloride contamination

- Because chloride is a very common ion, the contamination of the precipitation samples in lab is sometimes quite difficult to avoid.
- Considering the contamination with $SO3$ vapors, we avoided running the mineralization of soil or vegetal material with HCl or $HClO3$ simultaneous with chloride determination.
- All laboratory glasses used are washed before analysis with $HNO3$ 1:3.

a) Example of Ca, Mg contamination

- For metals analysis, aliquots of 100 ml samples were used, preserved in PE bottles at 4ºC in a refrigerator.
- Because we don’t have the possibility to use new bottle for each sample, we wash the bottles and reuse them.
- Once, we found very high concentrations of Ca and Mg in bulk precipitation samples and we couldn’t understand why.

- The problem was that we used some of the bottles of 100 ml to stock mineral waters samples, that had very high concentrations of Ca (more than 200 ppm) and Mg (more than 20 ppm).
- After being fixed on the polyethylene walls of bottles, the Ca and Mg ions diffused in the precipitation samples, because the bottles were not well enough washed.

- We found the good results by repeating the metal analysis from the original big bottle.
- We don’t use the aliquots bottles any more!

Thanks for your attention!