Analysis of heavy methals by ITP with pre-concentration focusing

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Mendelova zemědělská a lesnická univerzita v Brně

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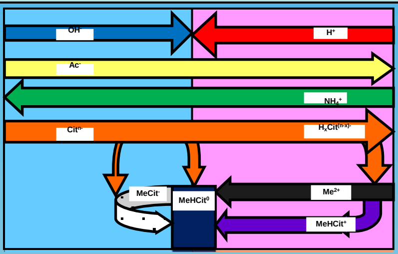
The capillary electrophoretic method for the focusing and selective pre-concentration of metal chelates with subsequent on-line ITP analysis was developed and verified. The selected ions of heavy metals (Na^+, Ca^{2+}, Mg^{2+}, Mn^{2+}, Cd^{2+}, Zn^{2+}, Ni^{2+}, Pb^{2+}, Cu^{2+}) were pre-concentrated from the mixture and analyzed. Focusing of metals was carried out in ligand field step gradient, which was realized by an addition of a convenient ligand agent to the regular stationary pH step gradient. The metal ions were continuously dosed into the column, where they were selectively trapped on the stationary ligand field step gradient in the form of non moving zones of citrate complexes with effective zero charge. After accumulation of detectable amount of analyte, the dosing was stopped and accumulated zones were mobilized to the analytical column, where they were analyzed e.g. by ITP method with conductivity or photometric detection. The reached increase in cLOD is ca. two orders of magnitude using acceptable dosing time 25min.

Principle

For the focusing of heavy metals in the ligand step gradient, the proper electrolyte was developed. It consists of two adjacent parts, alkaline, with a strong complexing power and acidic, with lower complexing power. Here, by influence of electric field, a stationary neutralization reaction boundary is created in the column.



The metals which exists in the acidic part preferably in the form of cations Me^{2+}, and in the alkaline part in the form of anionic complexes MeY^{n-} migrates towards the boundary and are here focused to the stationary zones of complexes with zero effective charge. If a metal is present in one electrolyte, e.g. in the acidic one, during the run is continuously dosed to the column, where is trapped on the boundary and accumulates here.

The accumulation is a function of dosing time, concentration and effective mobility of the metal-complex species. The selectivity of the focusing is given by pH of the electrolyte, by nature and concentration of the complexing /ligand/ agents. Metal ions creating lower and higher stability complexes pass through the boundary and/or migrate in the opposite direction and never reach the boundary. As an example a water analysis on presence of Pb and Cd is given, which it is not possible realized in common ITP mode.

Experimental

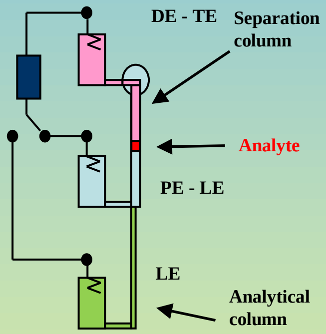
Electrolyte system

Alcaline focusing electrolyte:
LE/PE : 0,01M NH_4OH + 0,01M NH_4Ac
+ 0,002M sec. ammonia citrate
+ 1% polyethylenglycole, pH=9,24

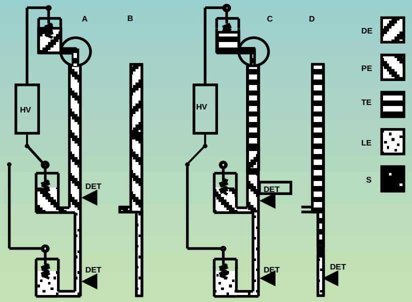
Acidic focusing electrolyte:
DE: 0,01M HAc + 0,01-0,001M NH_4Ac
+ 1% tritone X100, + sample metals.
pH 3,75-4,75.

ITP analytical electrolyte:
LE: 0,01-0,1M HAc + 0,01M NH_4Ac +
0,002M sec. ammonia citrate,
1% tritone X100, pH=4,75-4
TE: 0,03M HAc

Apparatus CS ITP analyser

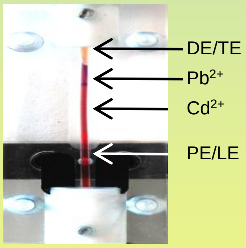


Analytical procedure

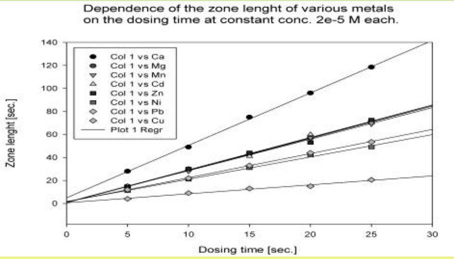


Focusing -A,B. Mobilization-C. Detection -D

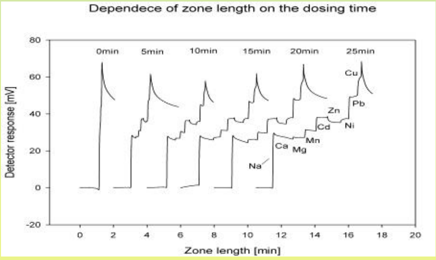
Focused zones of Cd^{2+}, Pb^{2+}, example of colored complexes with pyrogallol red



Dependence of the zone lenght on the dosing time (constant conc. of metal c= 2.10^{-5}).



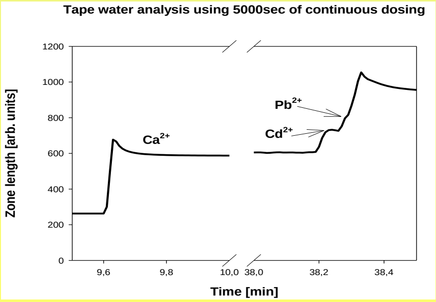
Analytical ITP record of dependence of the zone lenght on the dosing time (constant conc. of metal c= 2.10^{-5}).



Conclusion

Metal complexes of heavy metals were pre-concentrated, focused and analyzed using combination of ligand step gradient focusing method with subsequent mobilization and on-line e.g. ITP analysis. A continuous dosig technique was used for the lowering of detection limit, with the dosing time of 2000-3000 sec. a cLOD can be lowered by two orders of magnitude with favourable removing of sodium from the sample mixture. Such a low concentrated sample cannot by analysed by routine ITP analysis.

Tap water analysis with metod LSGF-ITP
5000 sec. preconcentration.



Acknowledgement

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