

Towards electrochemical measurements in the field of labile Cu and Pb in sedimentary particles

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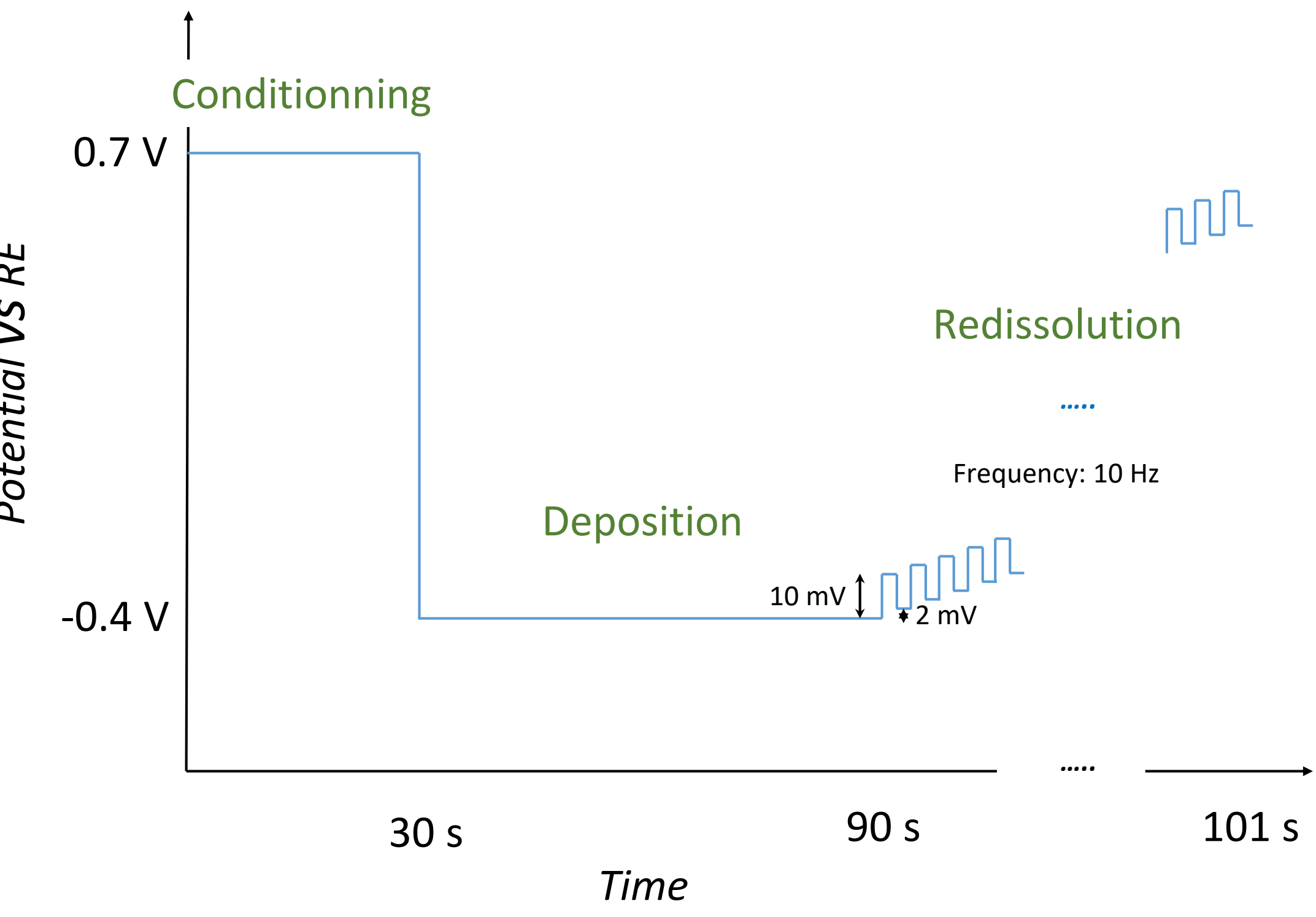
Introduction

Determining the overall sediment contamination by most metals is no longer an analytical problem in the laboratory. Plasma spectroscopy techniques allow reliable measurements by dissolving the sediment using concentrated acids or alkaline fusion. On-site, pXRF gives good results if the equipment is calibrated for the matrix under investigation and the samples are slightly pre-treated before analysis. This technique allows quick mapping, quick detection of hotspots, etc.

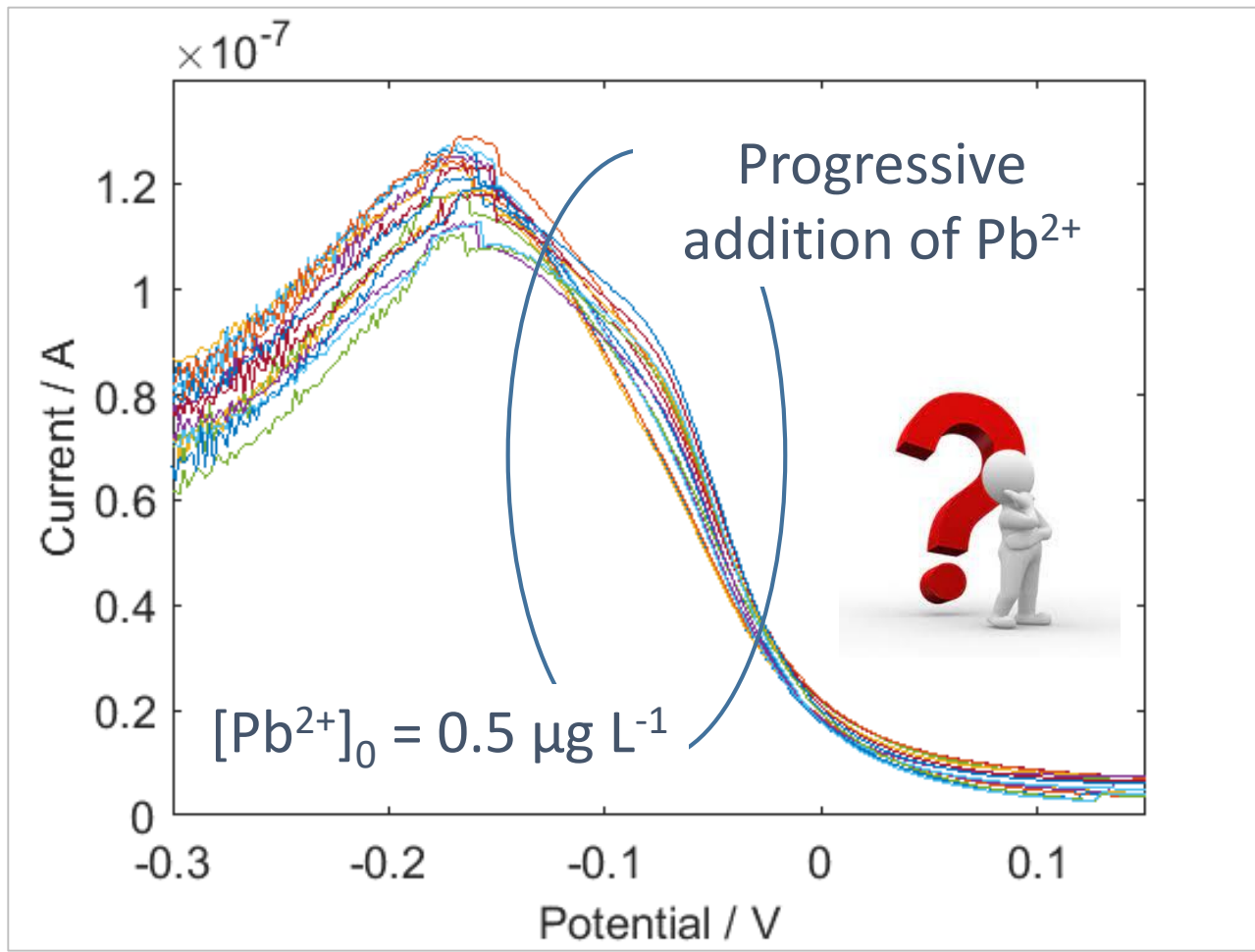
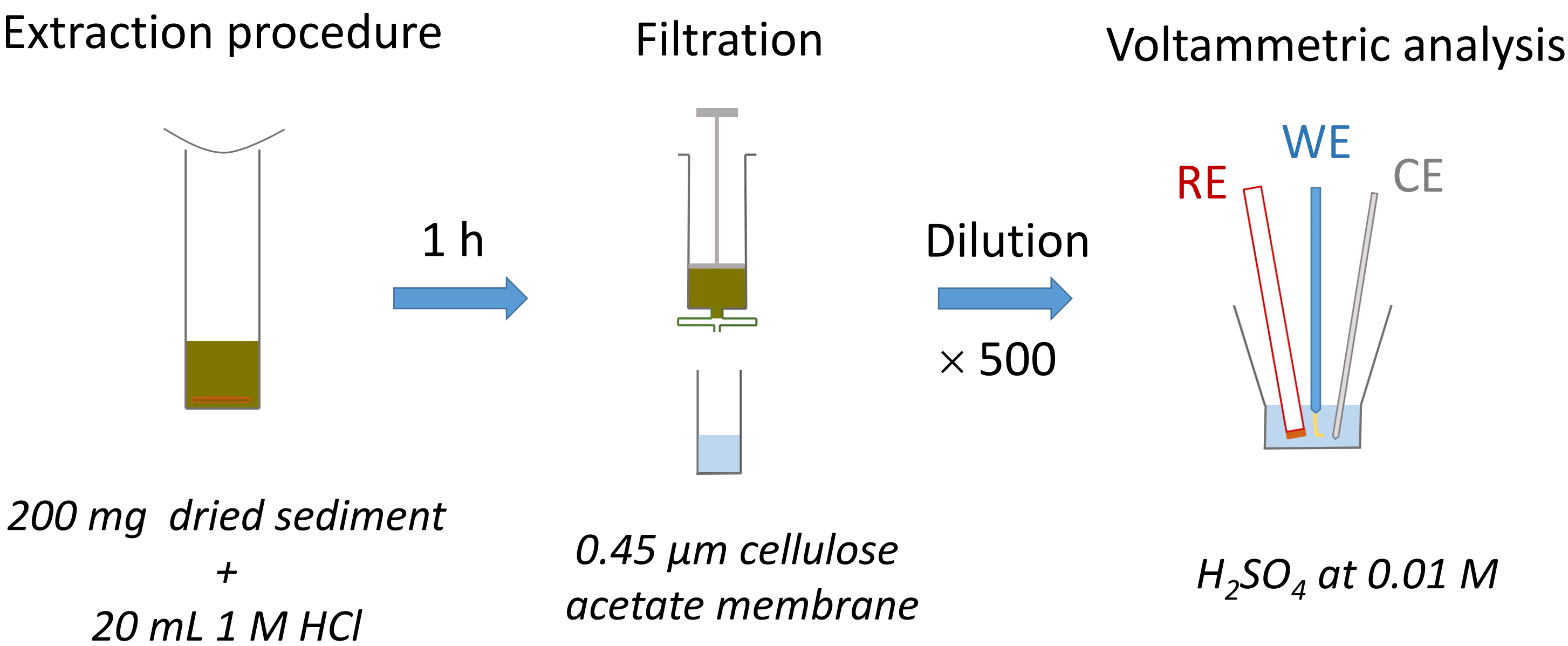
In this study, we have developed an electrochemical protocol for the measurement of easily extractable Cu and Pb, applicable in the field (particularly on sediment deposition sites) at a scale of one hour. This protocol can also be used for the measurement of these two metals in pore water, leakage water from dredged sediment... Indeed, pollution is not limited to the determination of total concentrations but must take into account the lability of the elements.

Material and methods

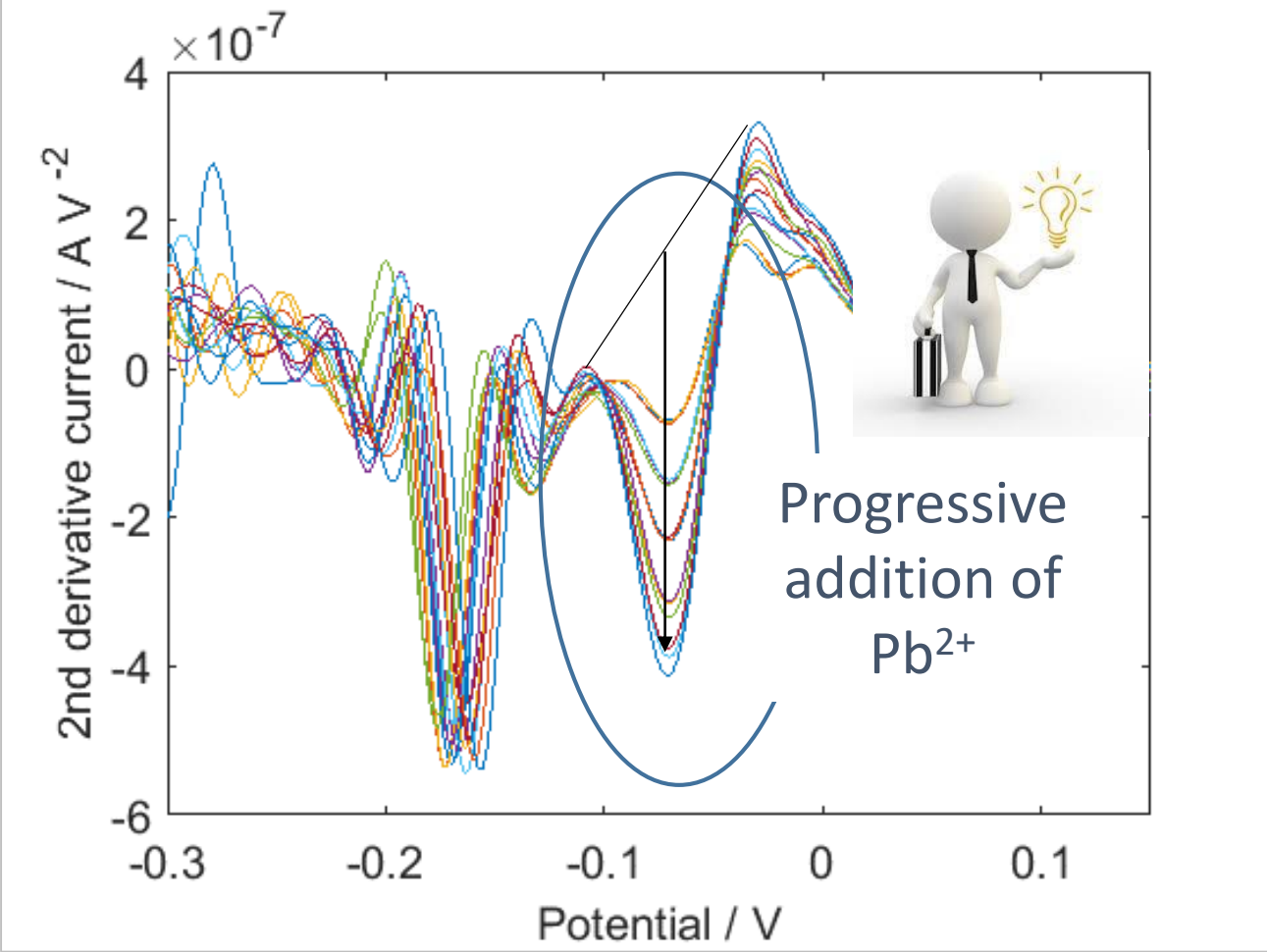
The device used is an Autolab type II potentiostat connected to the VA 663 stand and controlled by the GPES software. The IME 663 is used to manage the agitation of the solution present in the cell. Three different electrodes are used: a Ag/AgCl (3M KCl) reference electrode (RE), a micro gold wire as a working electrode (WE) and a platinum counter electrode (CE).



Voltammetric procedure
Anodic Stripping Square Wave Voltammetry (ASSWV)



Detection of Pb at a gold microwire electrode

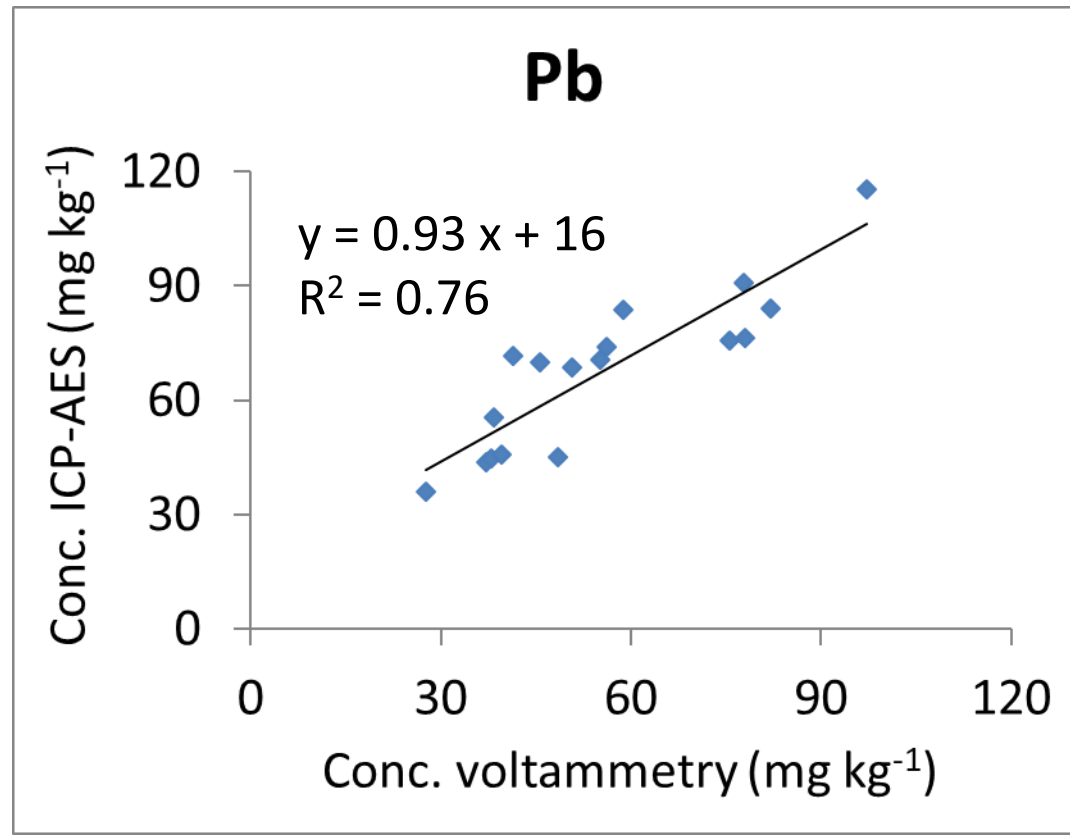
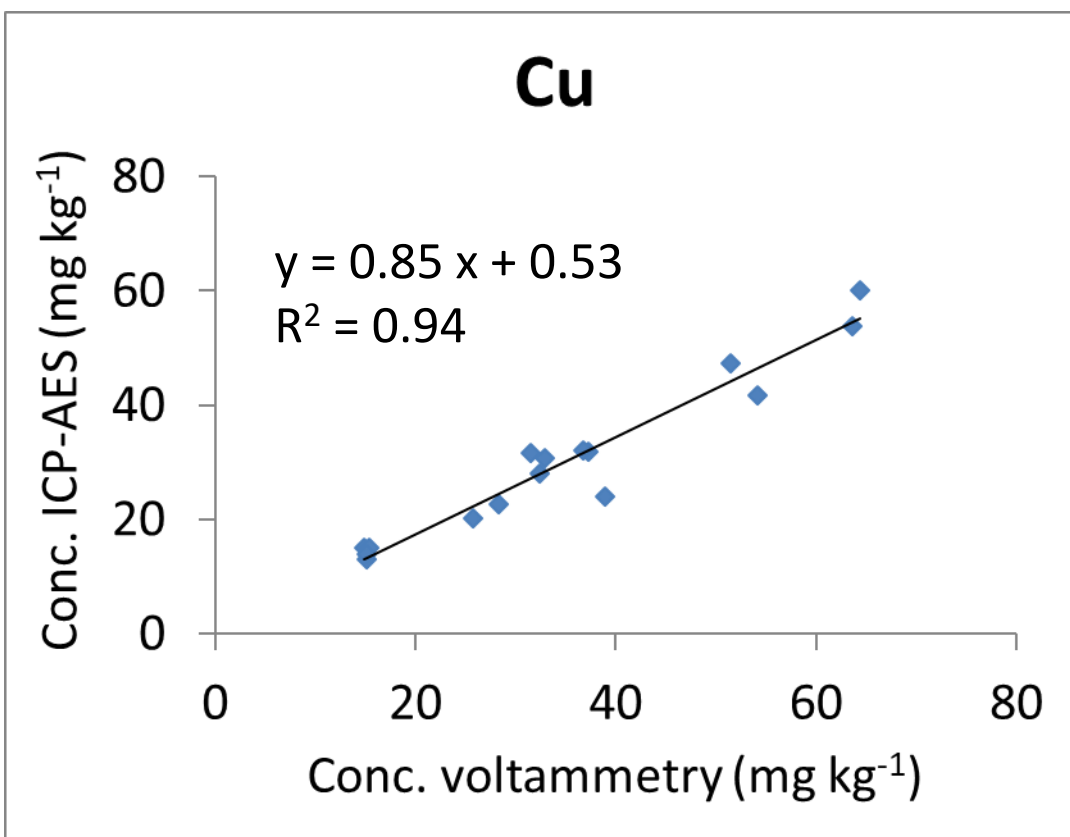


Second derivative current

Results and discussion

Performances (%)	Cu	Pb
Bias (B)	2	8
Repetability (R)	9	21
Linearity (L)	1	8
Zinc impact (ZI)	4	7
Total uncertainty (TU)	22	49

Values calculated from the French norm N° 17075 (ISSN 0335-3931)



Intercalibration between voltammetry and atomic emission spectroscopy

	Cu	Pb
Min conc. our method	25	25
Max conc. Our method	150	200
Threshold values of EU ¹	50-140	50-300
MDDEP 2014 (Québec)		
- housing & recreation	100	500
- industry & commercial	500	1000
Threshold values in Wallonie (Belgium) ²		
- agriculture & leisure	50	70
- industry & commercial	210	1500
values necessitating an intervention ²		
- agriculture & leisure	100	140
- industry & commercial	420	2300

¹ Dir 86/278/CEE

² <http://environnement.wallonie.be/legis/solsoussol/sol006.htm>

According to our analytical performances, the threshold values are in the range or above our maximal concentrations. It is therefore easy to dilute more our sample before analyses when the soil is highly polluted.

Conclusions

Given the results, voltammetric techniques are likely to obtain information on the mobility of metallic elements in sediments. However, pre-treatments are unavoidable, and the number of metals analyzed is very limited. Application in the field can be done using a miniaturized system but requires numerous manipulations (sampling, weighing, mineralization, filtration and dilution). This voltammetric approach could be more operational for aqueous matrices which are more diluted and do not require mineralization.