Green Electrochemistry: Determination of Heavy Metals at a Recycled Graphite Rod Electrode and Its Application for Trace Forensic and Environmental Analysis

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Outline of Talk

- The importance to study Pb levels
- Zinc-carbon batteries
- Fabrication of electrodes
- Cyclic voltammetric behaviour of Pb
- Anodic stripping voltammetry of Pb
- Trace determination of Pb in water
- Trace determination of Pb in dust wipe samples
- Conclusions

Removed from petrol, paint and plumbing

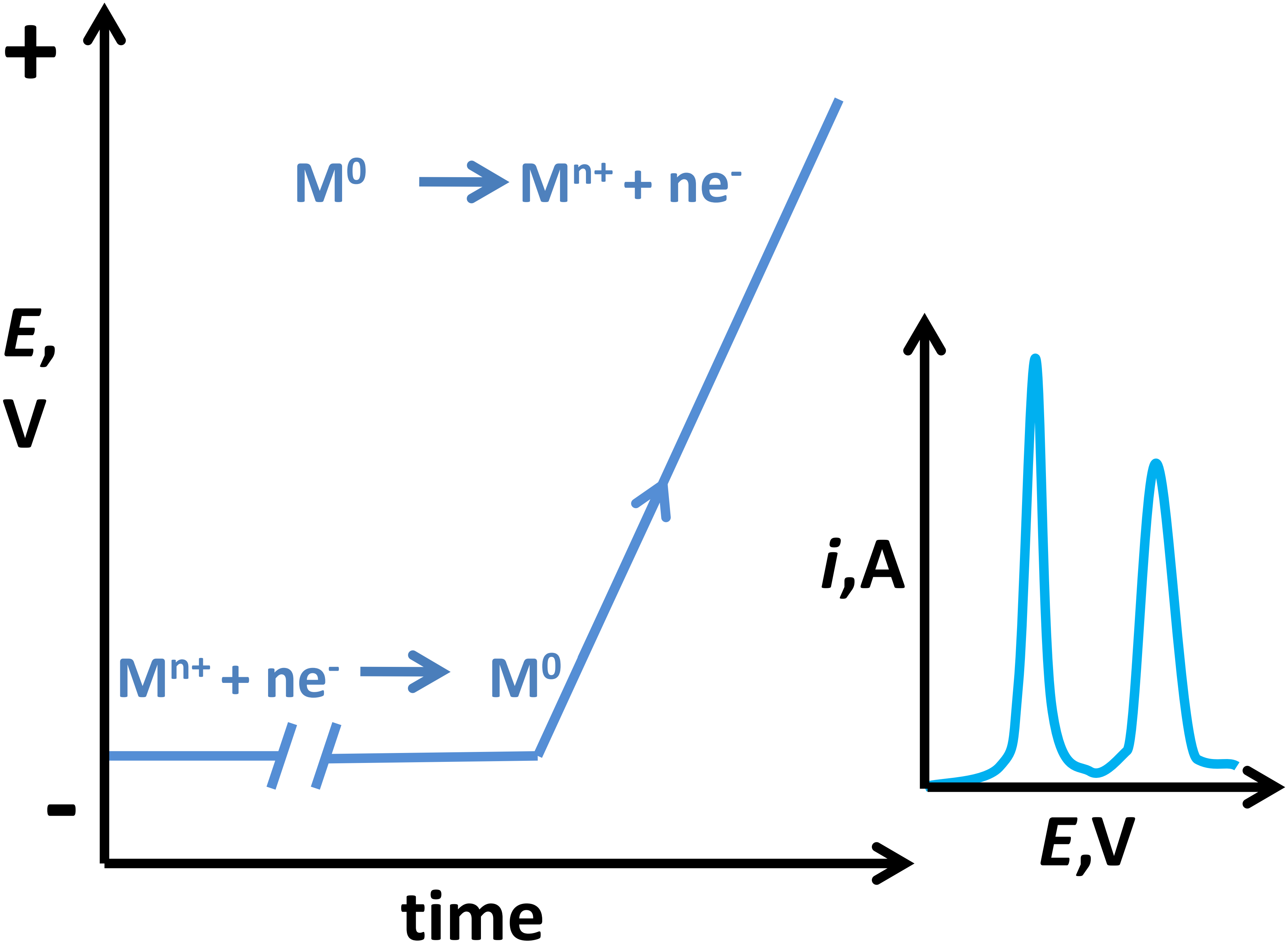
Legacy issues

Soil

Products from outside the EU

Figure 1. Disassembled zinc chloride cell (similar to zinc carbon cell). 1:entire cell, 2:steel casing, 3:zinc negative electrode, **4:carbon rod**, 5:positive electrode (Manganese dioxide mixed with carbon powder and electrolyte), 6:paper separator, 7:polyethylene leak proof isolation, 8:sealing rings, 9-negative terminal, 10-positive terminal (originally connected to carbon rod)

Cross-section of a zinc–carbon battery.


+
E, V
-
$M^0 \rightarrow M^{n+} + ne^-$
$M^{n+} + ne^- \rightarrow M^0$
time
i,A
E,V

Comparative Techniques

- Detection limits
  - Lower than many much more expensive/resource hungry techniques:
  - ICP AES 50 ng/mL
  - Flame AAS 10 ng/mL
- Anodic stripping voltammetry generally sub ng/mL. Really a factor of how good a quality your reagents are.

AAS, GFAAS, ICP or ICP-MS? Which technique should I use? An elementary overview of elemental analysis, Thermo Elemental, http://www.thermo.com/eThermo/CMA/PDFs/Articles/articlesFile_18407.pdf accessed 18/2/14.

Cyclic voltammetry of Pb at GRE

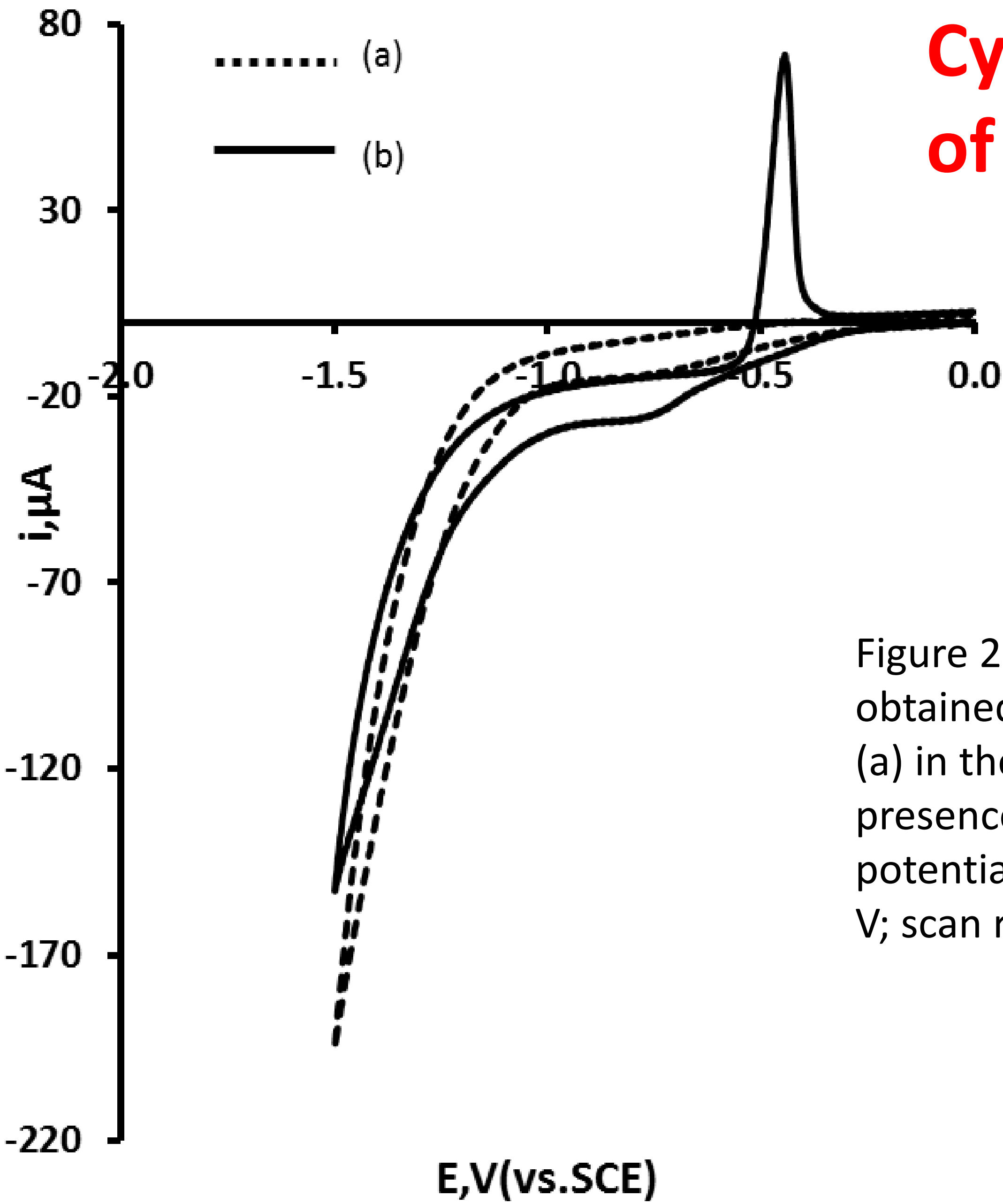
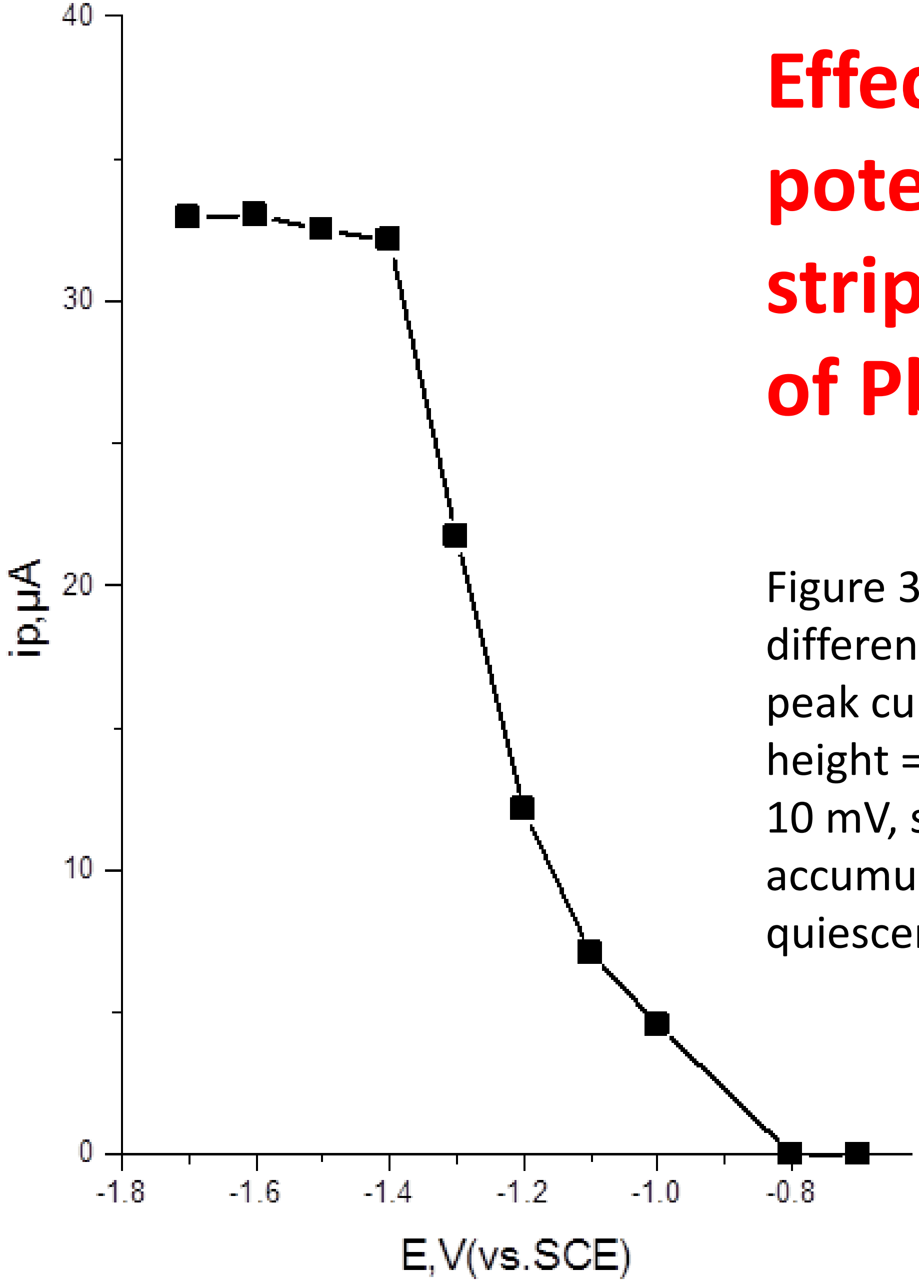


Figure 2. Typical cyclic voltammograms obtained with GRE for 1.0 M acetic acid, (a) in the absence of and (b) the presence of 0.1 mM Pb. Initial and finial potential: 0.0 V; switching potential −1.5 V; scan rate 50 mV/s.



Effect of deposition potential on the anodic stripping voltammetry of Pb at GRE

Figure 3. Effect of deposition potential on differential pulse anodic stripping voltammetric peak current. Voltammetric conditions: pulse height = 100 mV, pulse width = 50 ms, step height = 10 mV, step width = 0.2 s. 0.1 mM Pb^{2+} 45 s accumulation time with stirring followed by 15 s quiescent in 4% v/v acetic acid.

Effect of accumulation time on the anodic stripping voltammetry of Pb at GRE

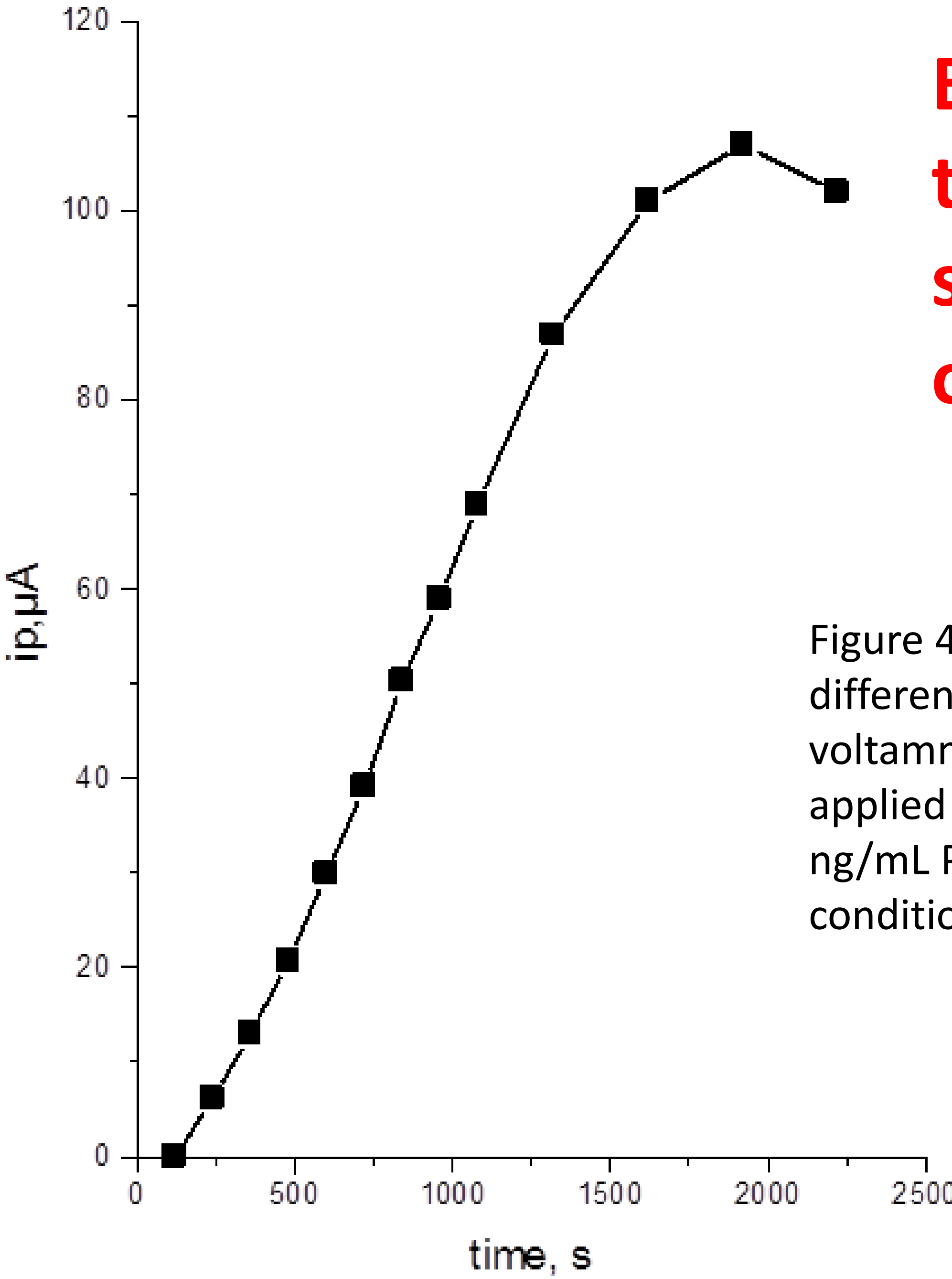
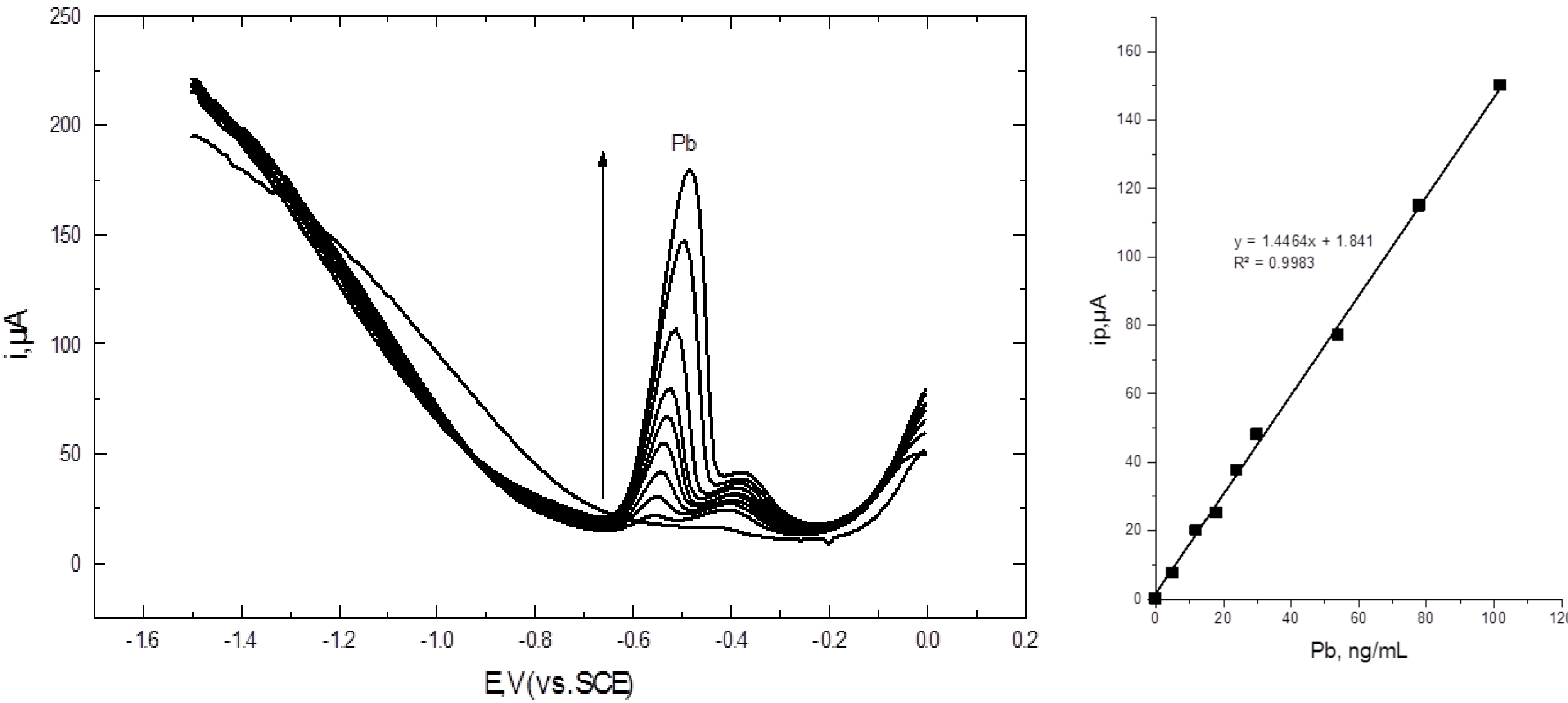


Figure 4. Effect of accumulation time on the differential pulse anodic stripping voltammetric peak current obtained for an applied potential of -1.5 V (vs. SCE) for a 108 ng/mL Pb^{2+} solution. Other voltammetric conditions as Figure 3.

Effect of concentration on the anodic stripping voltammetry of Pb at GRE



Linear range = 2.4 µg/L to 110 µg/L (R^2 = 0.999, 1.45 µA/ng/mL).

Theoretical detection limit of 0.3 ng/mL was calculated.

Figure 5. Effect of Pb concentration on anodic stripping peak. Accumulation time 1100 s, other voltammetric conditions as figure 4.

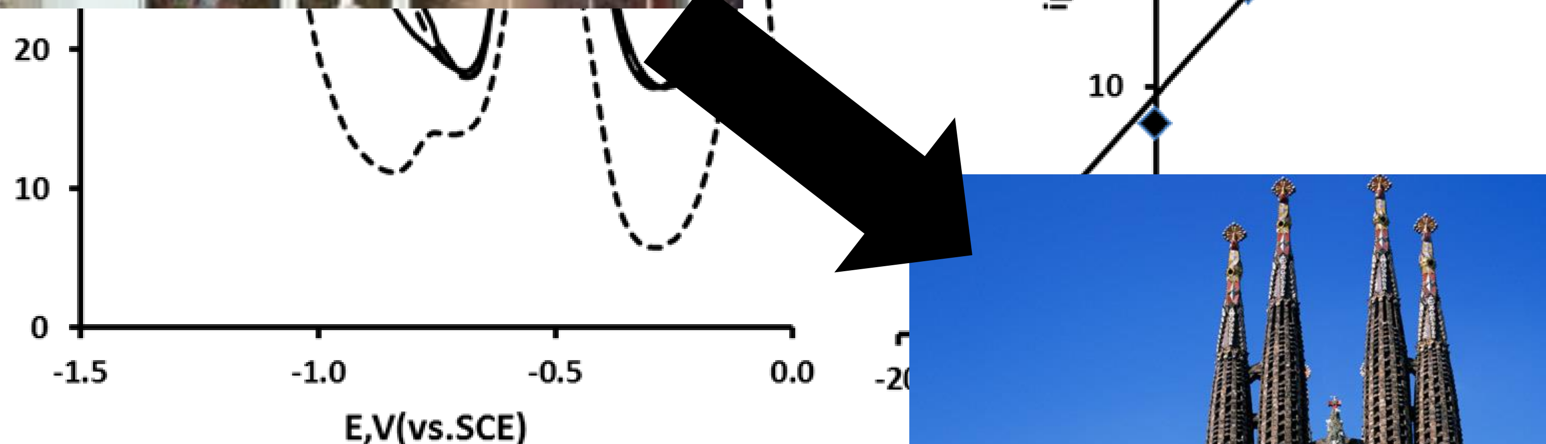
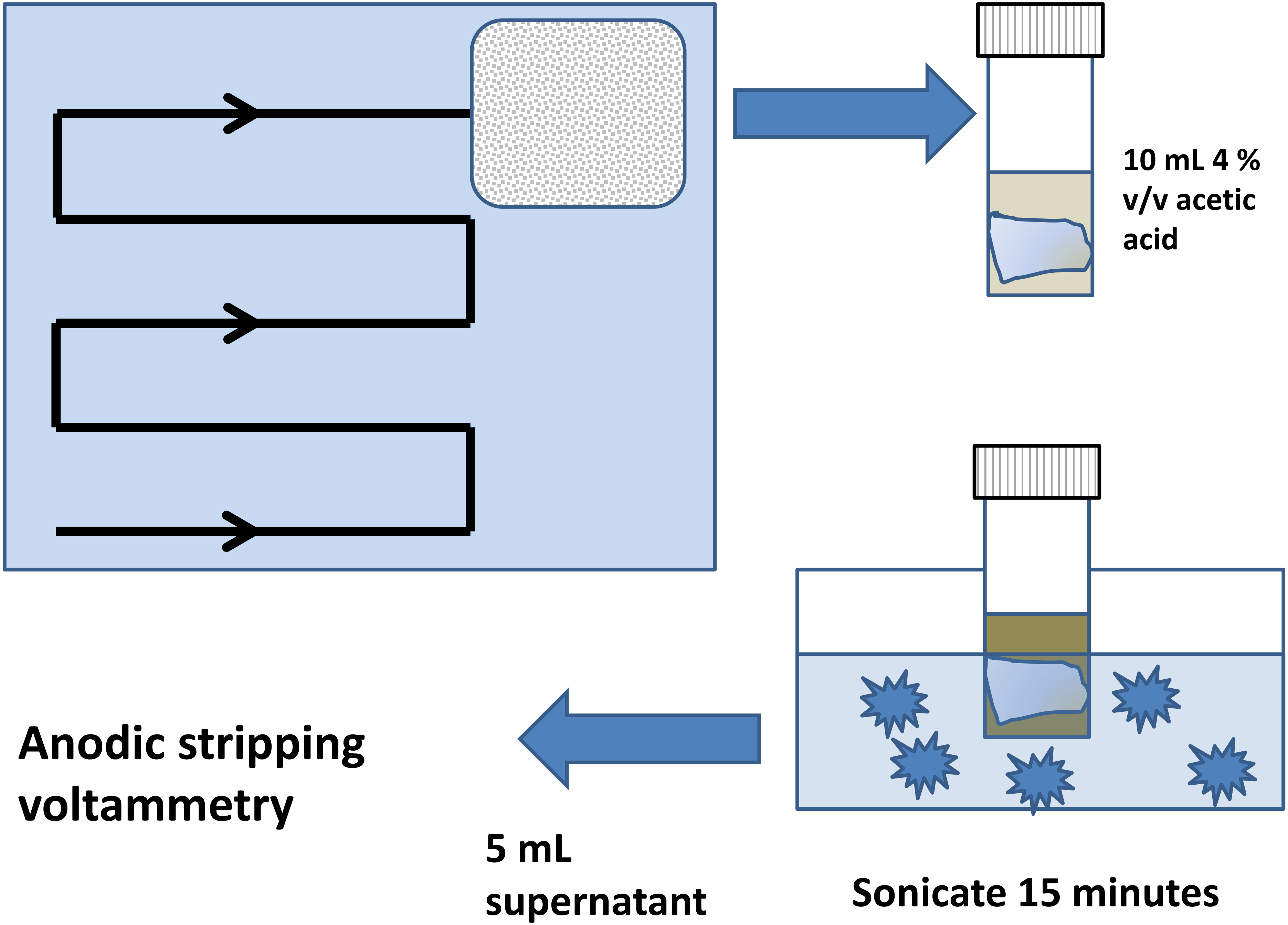
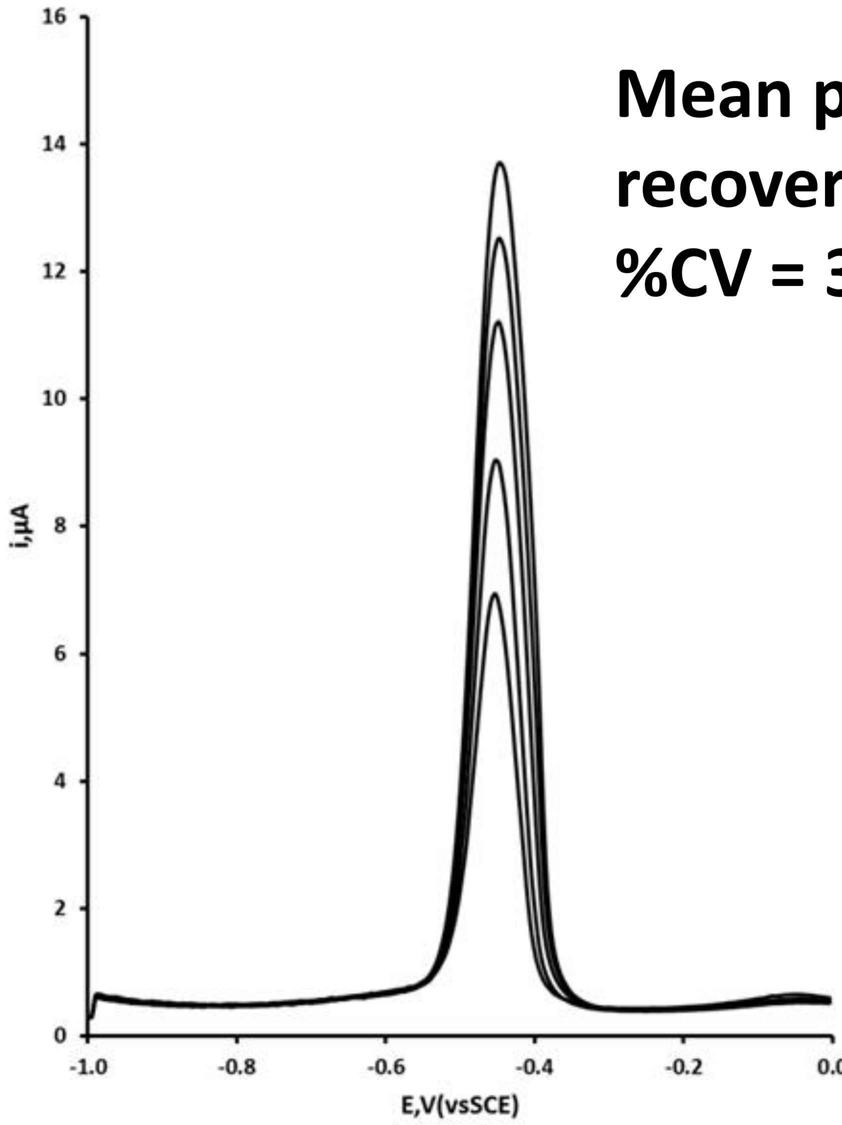


Figure 6. (a) Differential pulse voltammograms ob
of Pb made to water sample fortified with 16 ppb
plot.

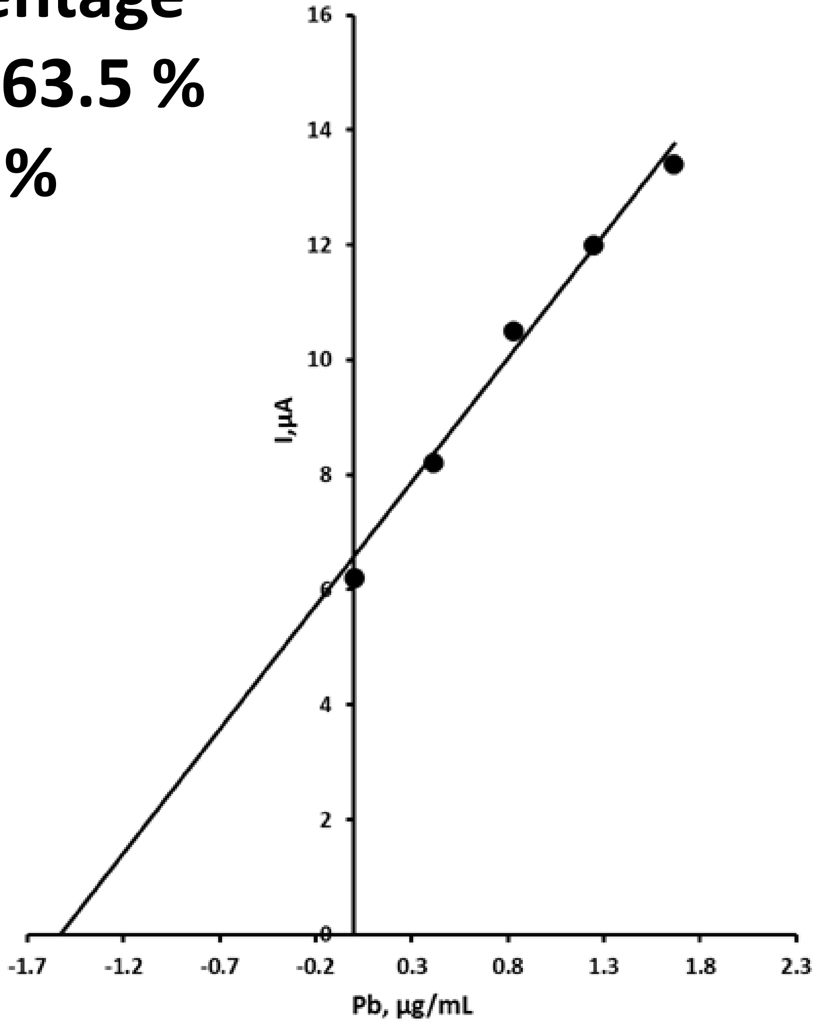

10 mL 4 %
v/v acetic
acid
Anodic stripping
voltammetry
5 mL
supernatant
Sonicate 15 minutes

Determination of Pb in dust wipe samples by anodic stripping voltammetry of Pb at GRE



Mean percentage recovery = 63.5 %

%CV = 3.26 %



Conclusions

- Demonstrated the possibility to make electrodes from zinc-carbon batteries
- Explored their electrochemical behaviour by cyclic voltammetry
- Optimised the conditions necessary for the trace determination of Pb
- Shown the possibility to determine trace Pb concentrations in real samples
- Enhancing links between research and teaching

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Thank You

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