Trace Analysis of Concentrated Sulphuric Acid and Zinc Sulphate by Flow-Through Coulometry

Ernest Beinrohr, Pavol Beinrohr and Stanislava Hlubikova

Department of Analytical Chemistry

Slovak Technical University, Bratislava

ISTRAN, s.r.o., Bratislava

Tasks for analytical chemists:

Raw materials

Products

- Bulk analysis
- Surface analysis

Wastes

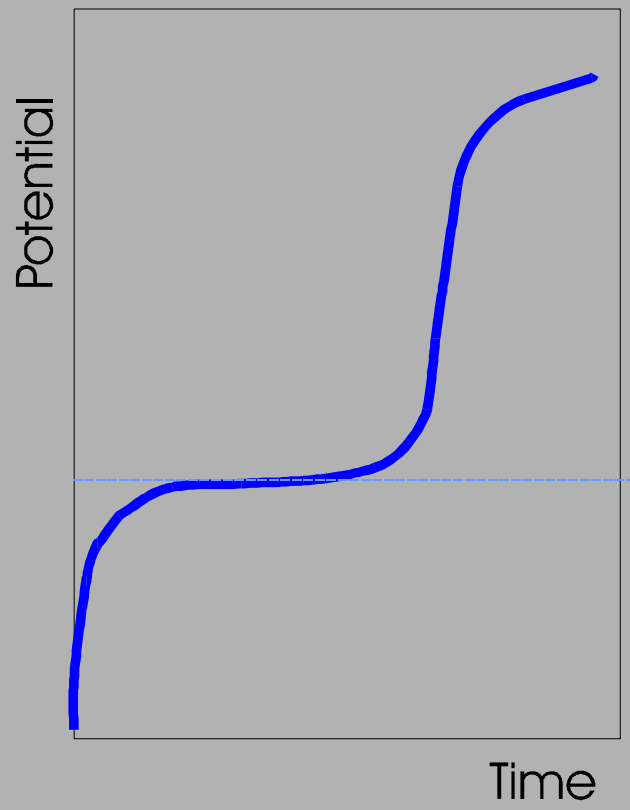
Bulk analysis

Requirements:

- Robust system
- Automation
- Low cost/analysis

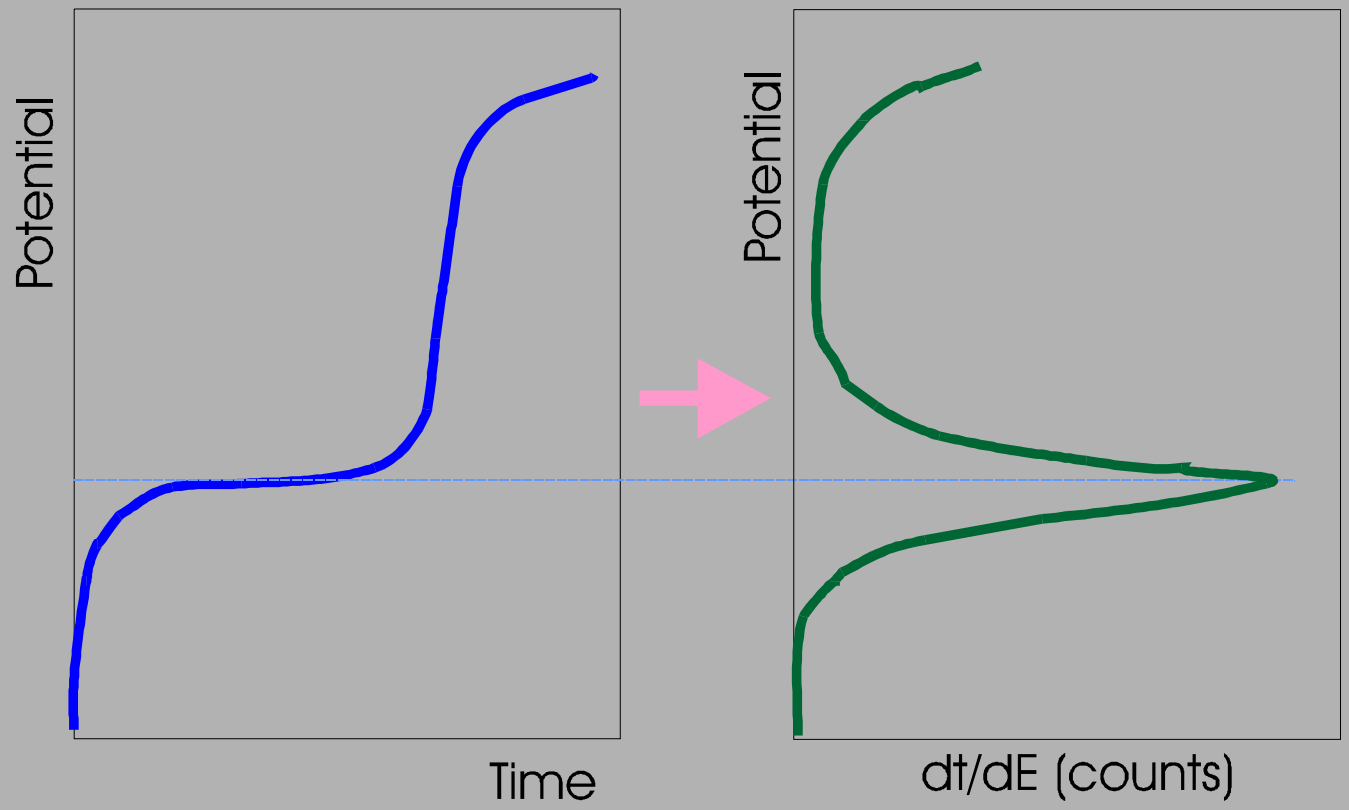
Theory

Chronopotentiometry

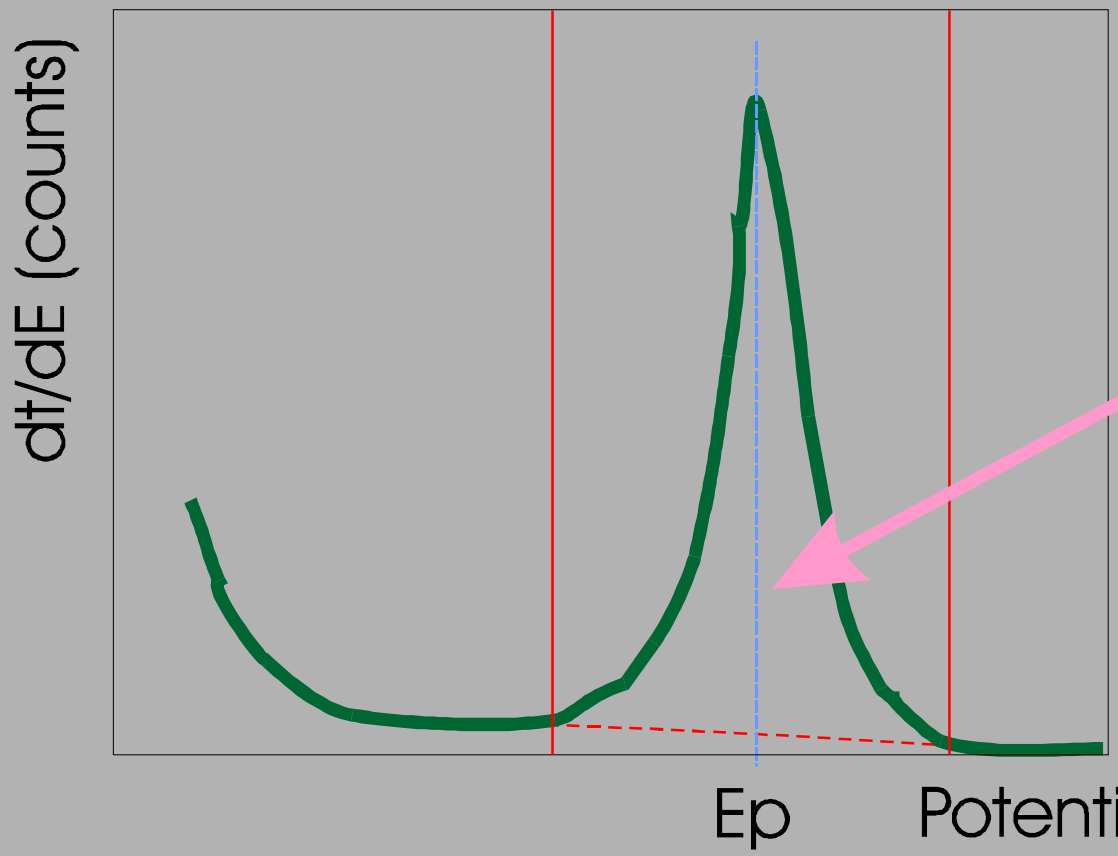


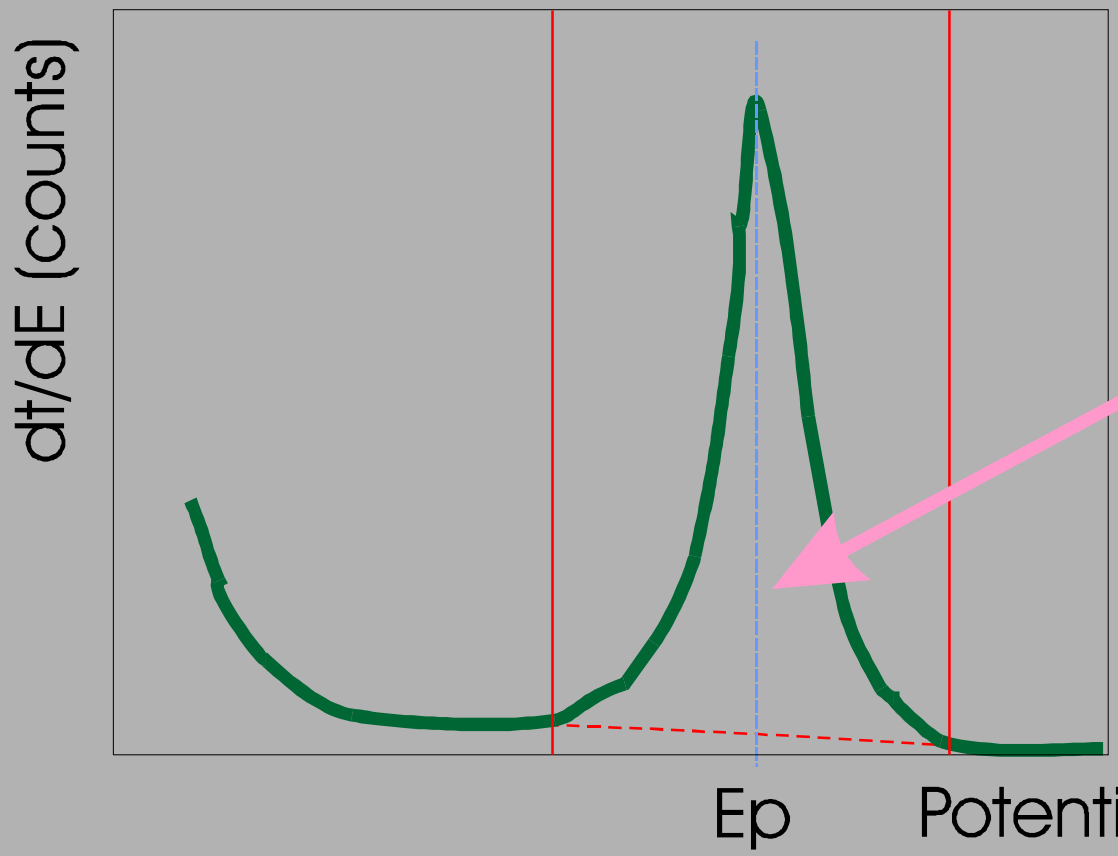
$$Pb^{0} - 2\ e^{-} \Rightarrow Pb^{2+}$$

Chronopotentiometry



$$Pb^{0} - 2\ e^{-} \Rightarrow Pb^{2+}$$


dt/dE (counts)
Tau (Peak area)
Ep
Potential

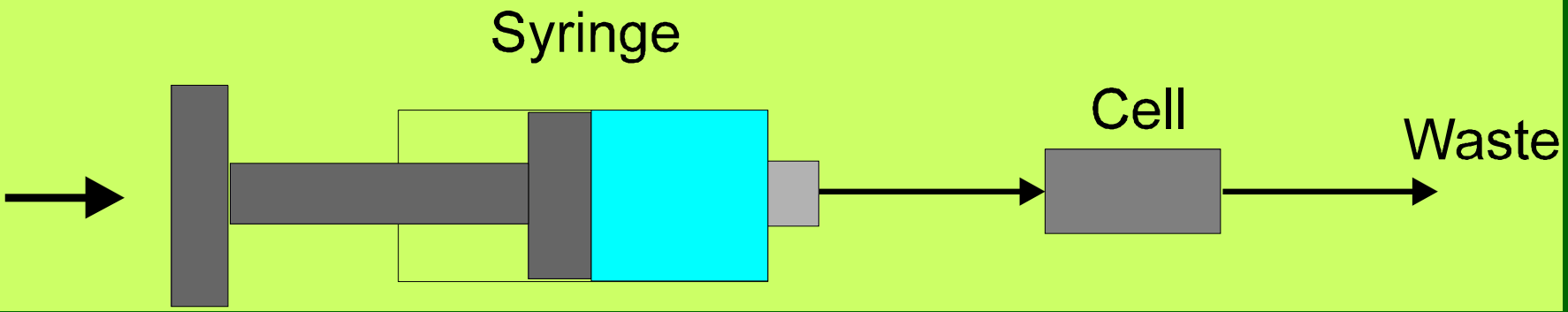


$$Tau = R \; z \; F \; c \; V \; / \; I$$

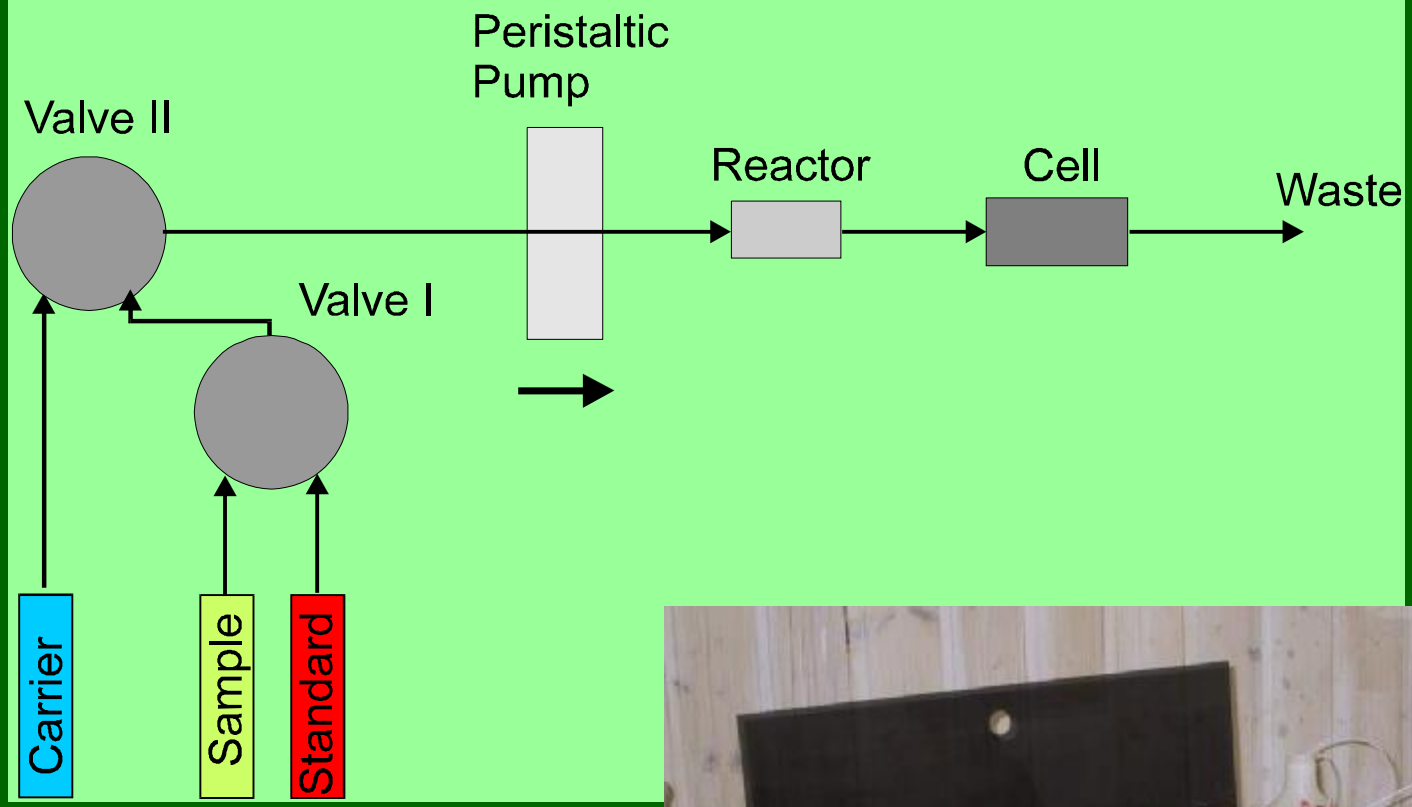
Experimental

The cell and electrode

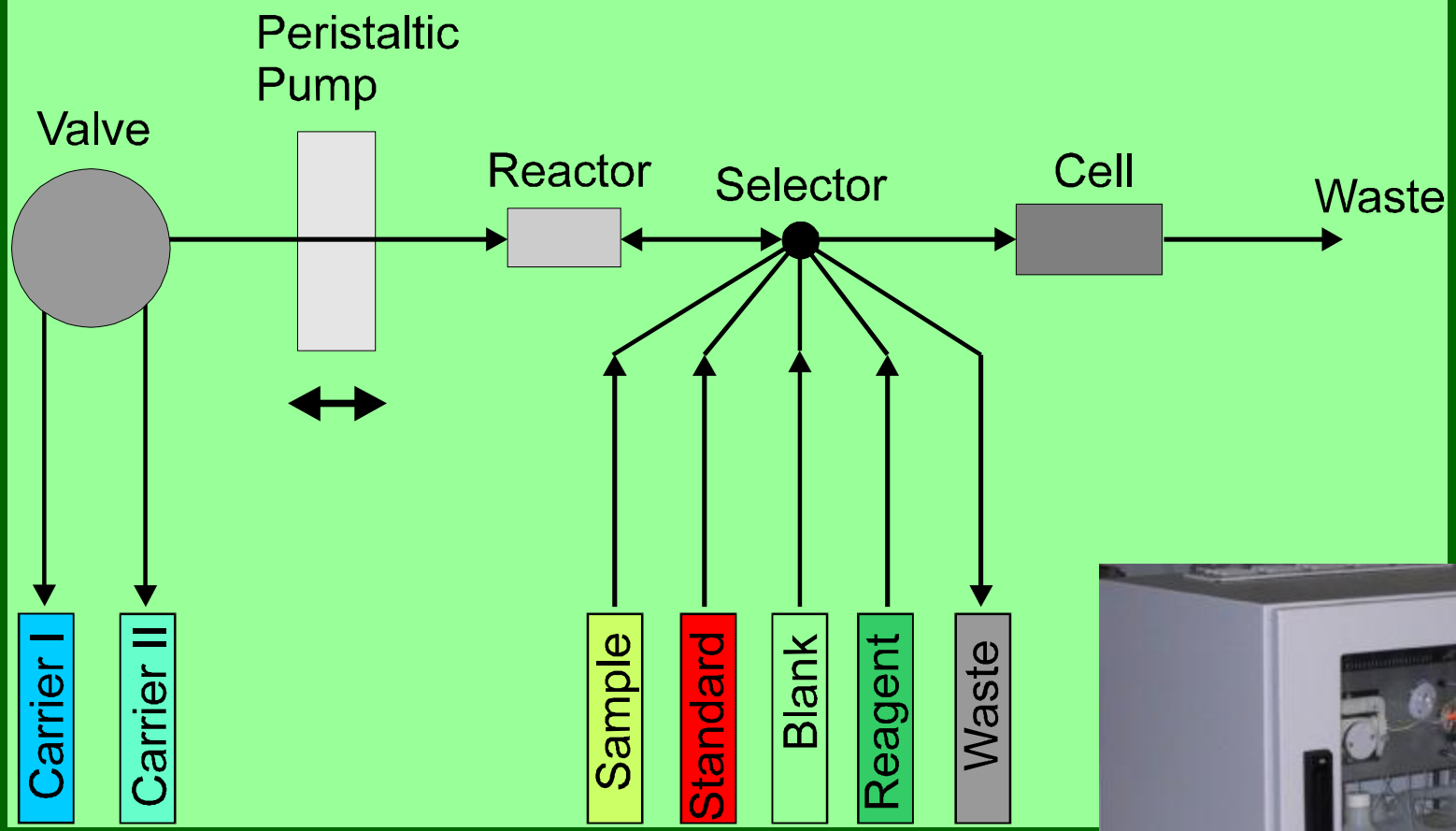
EcaSystem "MIA"



EcaSystem "FIA"



EcaSystem "SaFIA"

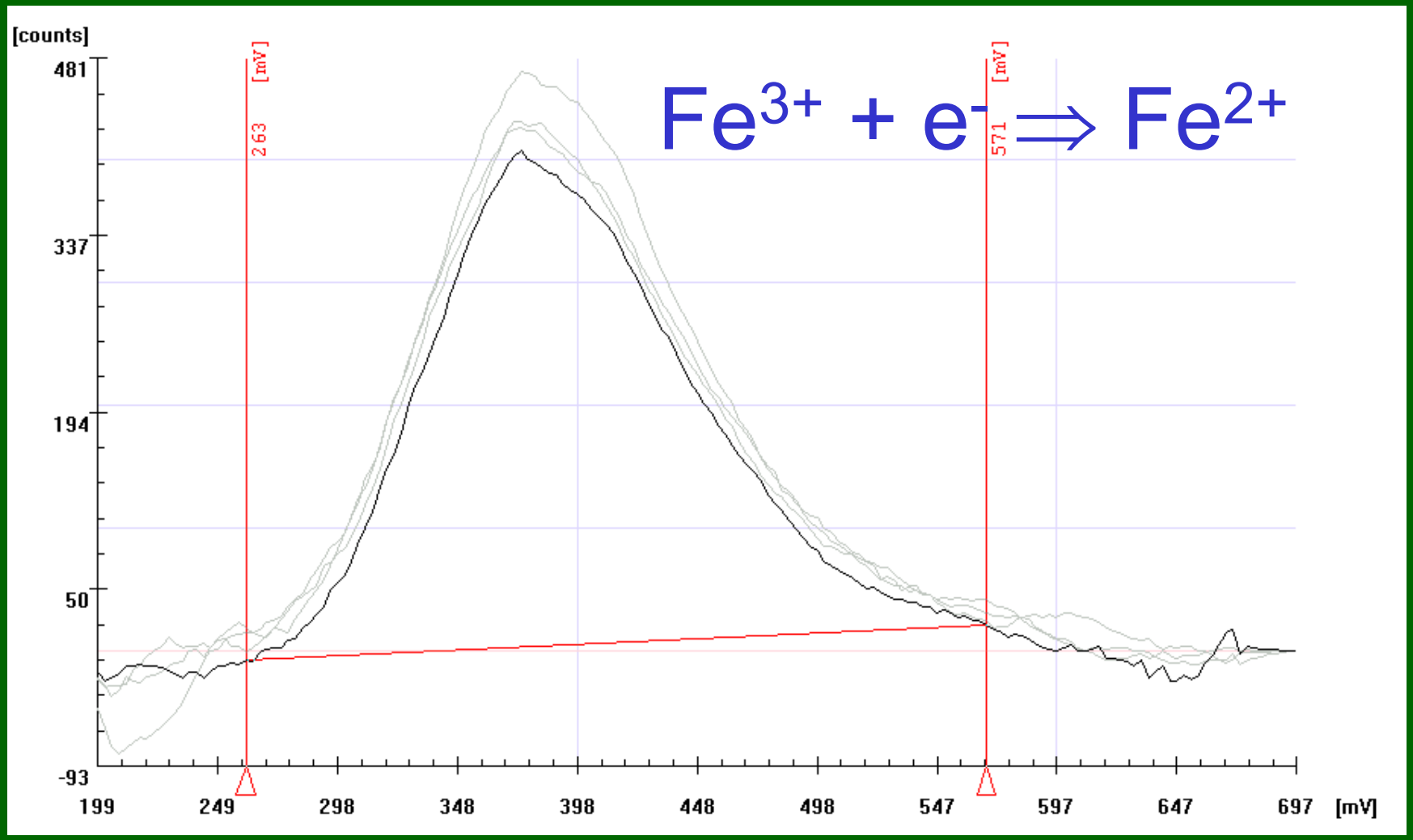


Results

Fe in sulphuric acid

Parameters:

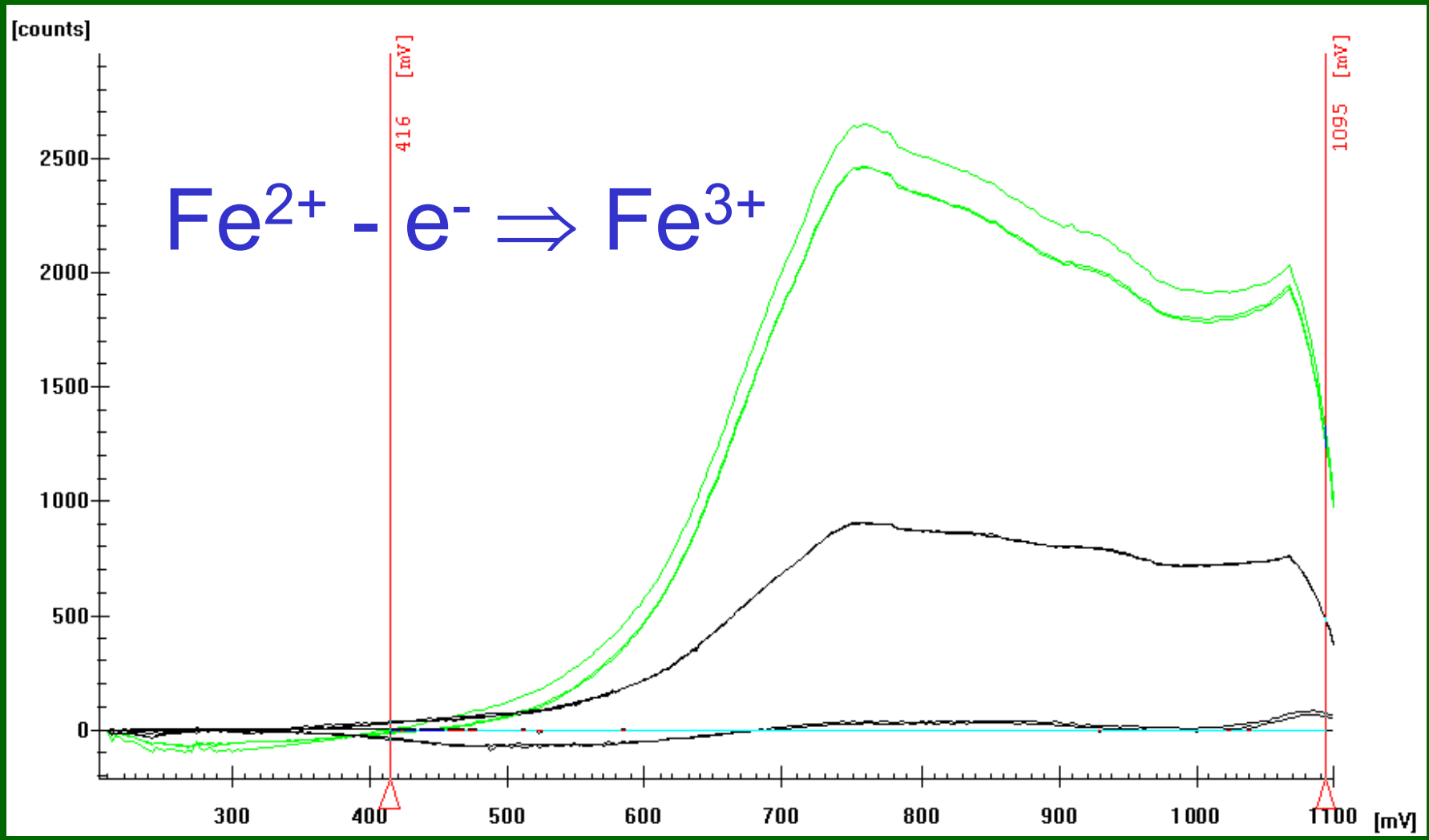
Electrode:	microporous E-53C
Electrolyte:	diluted HCl
Method:	in-electrode coulometric titration (reduction)


[counts]
481
337
194
50
-93
263 [mV]
571 [mV]
$Fe^{3+} + e^- \Rightarrow Fe^{2+}$
199
249
298
348
398
448
498
547
597
647
697
[mV]

Fe in zinc sulphate

Parameters:

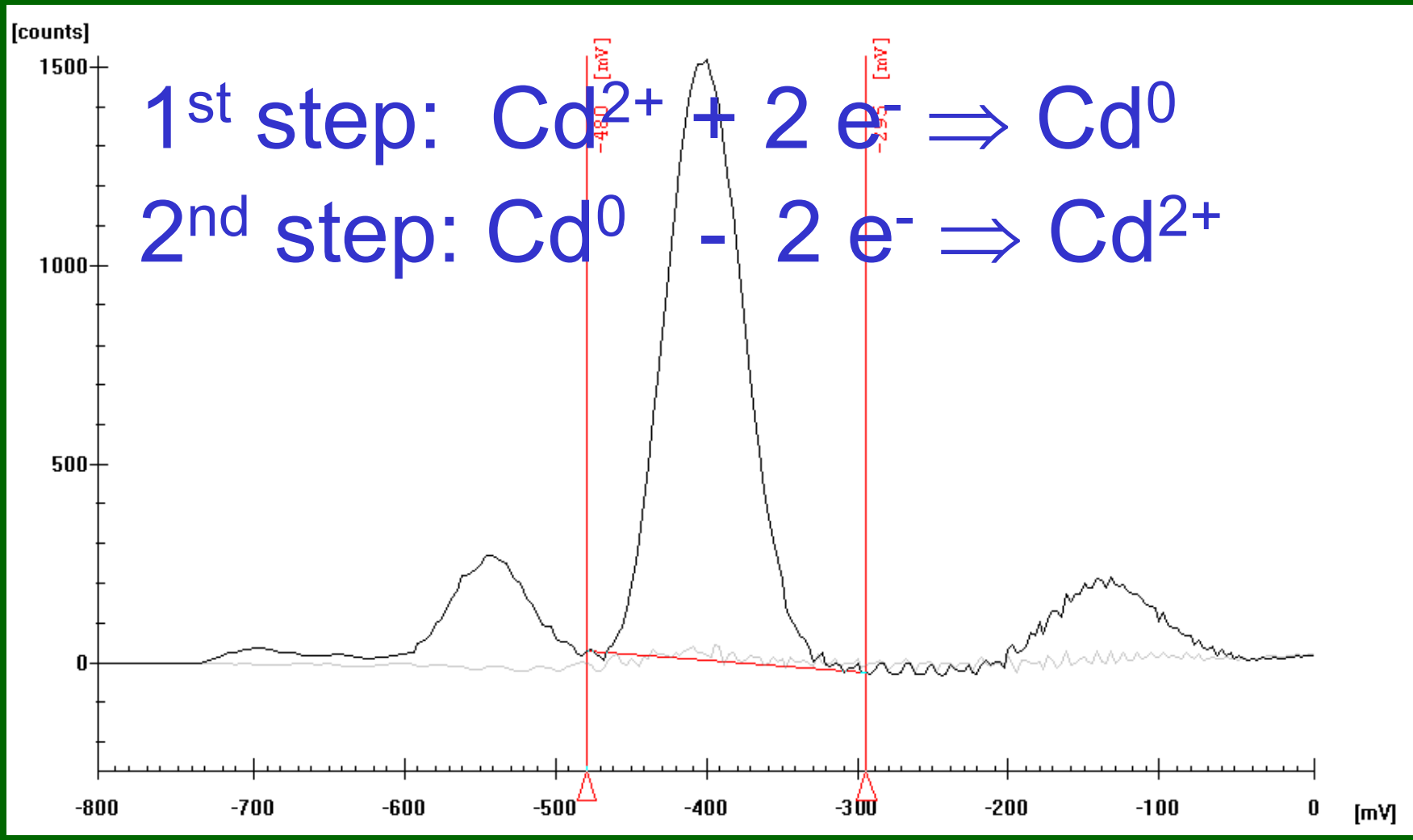
Electrode:	macroporous E-104N
Electrolyte:	diluted HCl
Method:	in-electrode coulometric titration (oxidation)


[counts]
2500
2000
1500
1000
500
0
416 [mV]
1095 [mV]
$Fe^{2+} - e^{-} \Rightarrow Fe^{3+}$
300
400
500
600
700
800
900
1000
1100
[mV]

Cd, Pb, Cu in zinc sulphate

Parameters:

Electrode:	macroporous E-104L
Electrolyte:	diluted HCl
Method:	stripping coulometry

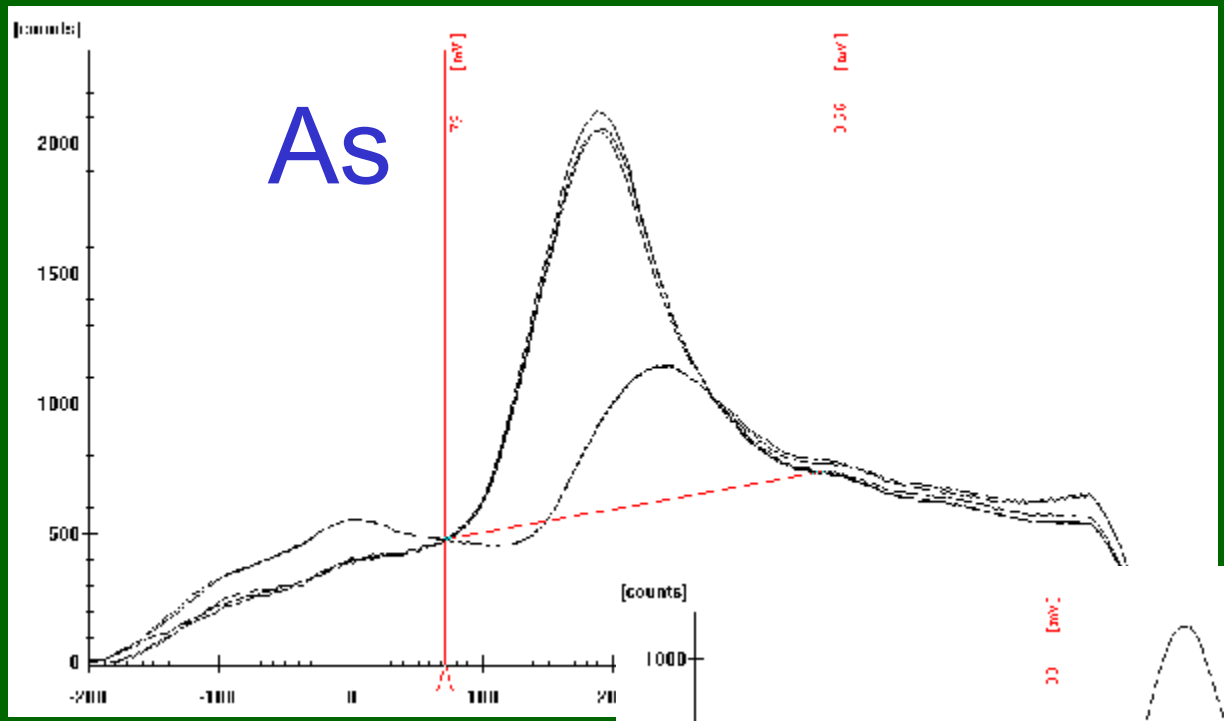

[counts]
1500
1000
500
0
-800
-700
-600
-500
-400
-300
-200
-100
0
[mV]
-480 [mV]
-595 [mV]
1st step: Cd2+ + 2 e- ⇒ Cd0
2nd step: Cd0 - 2 e- ⇒ Cd2+

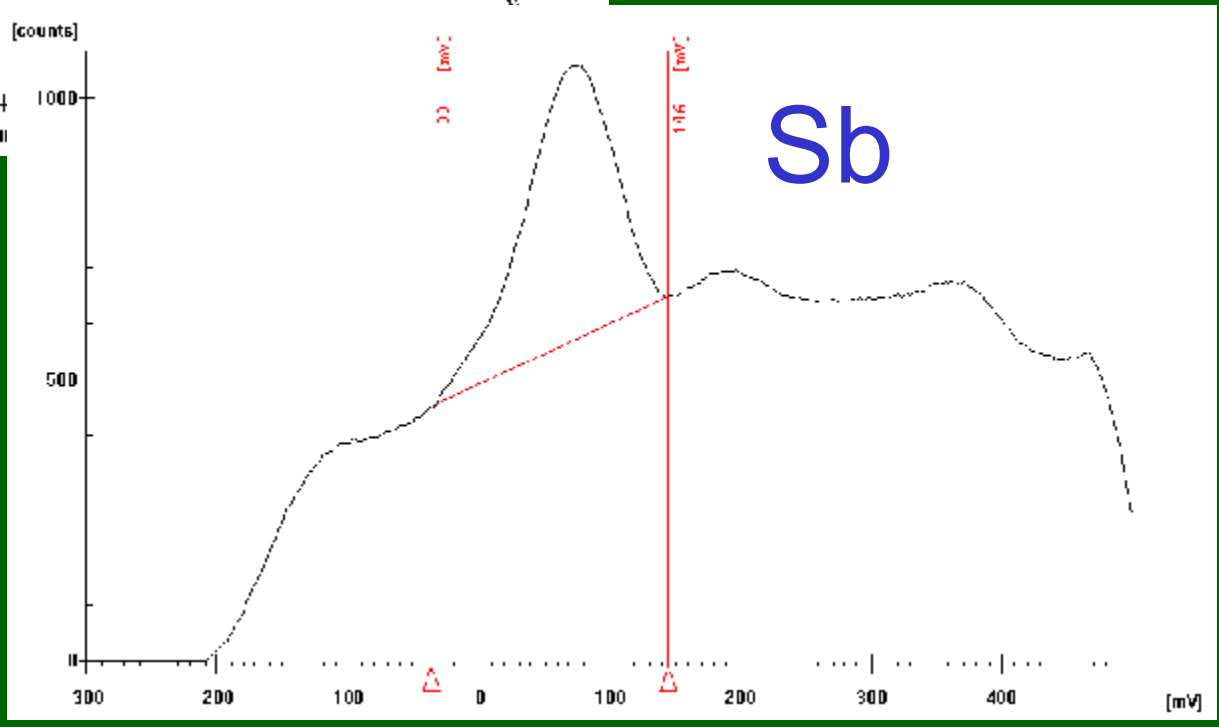
Cd Pb Cu

As, Sb in zinc sulphate

Parameters:

Electrode:	E-T/Au and E-CA/Au
Electrolyte:	diluted HCl
Method:	stripping coulometry

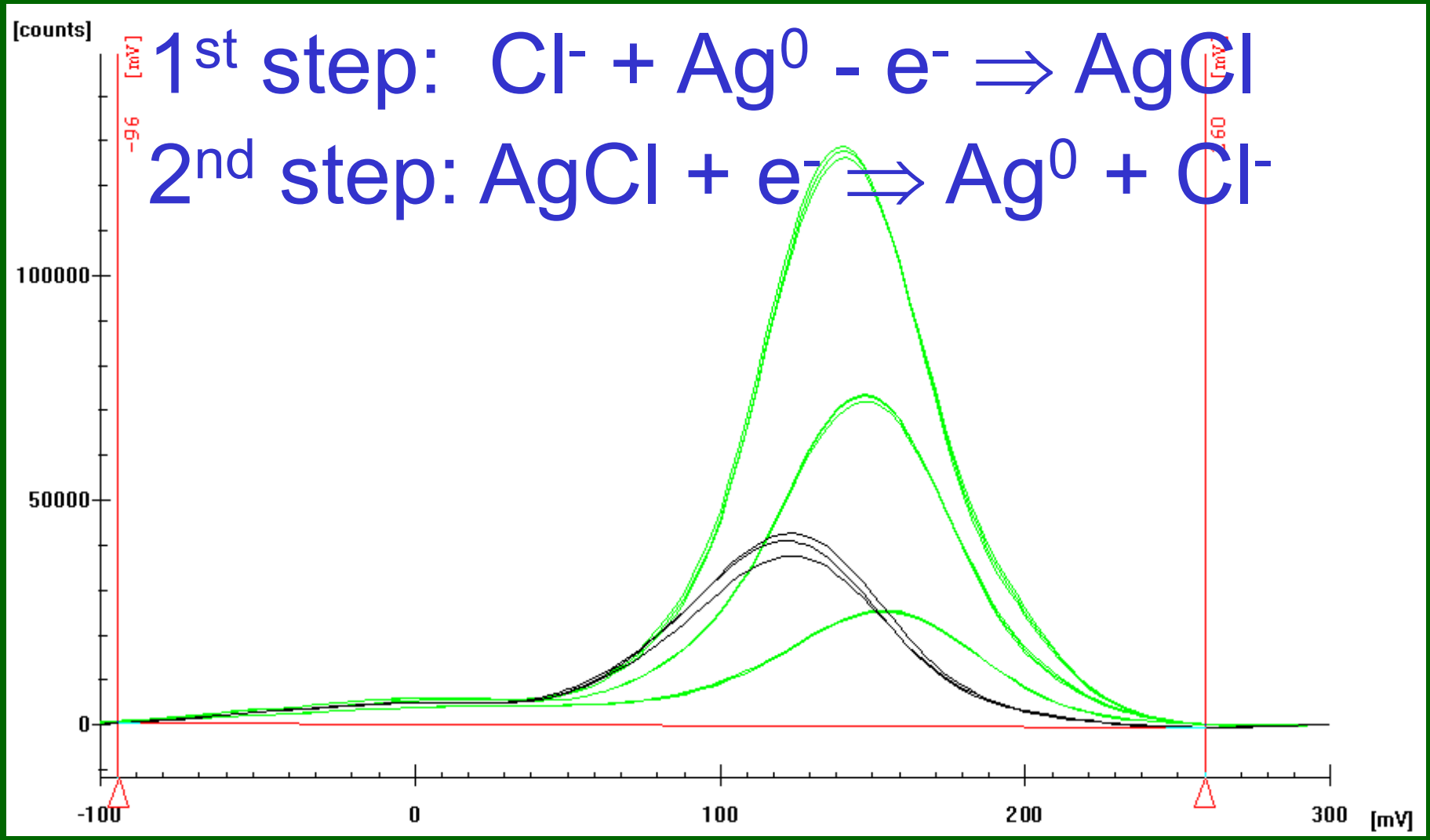

As
[counts]
2000
1500
1000
500
0


Sb
[counts]
1000
500
0
300
200
100
0
100
200
300
400
[mV]

Chlorides in zinc sulphate

Parameters:

Electrode:	E-CA/Ag
Electrolyte:	diluted HNO_3
Method:	coulometric titration


1st step: Cl- + Ag0 - e- ⇒ AgCl
2nd step: AgCl + e- ⇒ Ag0 + Cl-
[counts]
[mV]
-96
[mV]
-60
100000
50000
0
-100
0
100
200
300
[mV]

Conclusions:

Fe in sulphuric acid can be measured down to 0.1 mg/L

Cd, Pb, Cu, As, Sb, Mn in zinc sulphate can be measured down to 0.01 mg/L

Chlorides in zinc sulphate can be measured in the range of to 1 to 1000 mg/L

and more …

Metals:

Fe, Ni, Cr, Pb, Cd, Bi, Hg …

Semimetals:

As, Se, Sb

Non-metals:

S^{2-}, Cl^{-}, NO_3^{-}, PO_4^{3-} …

Organics:

EDTA …

Thank you