Nickel plating bath

Electroless nickel
Electroless nickel is the most important catalytic nickel plating process working without electrical supply.
Main components:
- Nickel sulphate
- Sodium hypophosphite
- Chelating agents (mono or di or tri carboxylic satured organic acids, amino acids, ethylenediamines o pyrophosphates)
- Stabilisers and inhibitors (Compounds based on: Se, Te, AsO_2^-, IO_3^-, MoO_4^{2-}, Sn^{2+}, Pb^{2+}, Sb^{3+}, unsatured organic acids and sulphur, like thiourea)
- pH adjusters (NH_4^+)

Electroplating bath
Two similar processes for electroplating nickel bath are commonly used: Watt process and Nickel sulfamate.

Nickel Watt
Main components:
- Nickel chloride
- Nickel sulphate
- Boric acid

Nickel sulfamate
Main components:
- Nickel chloride
- Nickel chloride sulfamate
- Boric acid

Main additives for both the bath: anti stress agents (sources of sulphur like: saccharin, p-toluene sulfonamide, m-benzenedisulfonate, 1,3,6 sodium naphtalen trisulfonate), levelling agents (i.e. 2 butyne 1,4 diol), sparkling agents, wetting agents and penetrating agents.

Pollutants
In these bath the following unwanted metals are analysed:
Iron (> 200 mg/l) enhances internal stress of the nickel layer.
Copper and zinc (> 10 mg/l)reduce ductility.
Aluminium (> 6 mg/l) generates burned corners effects.
Chromium and lead: reduces hardness and the efficiency of the current transport, even if at low concentrations.

Analysis of Nickel

Pour 10 ml of 0.1 M KNO_3 (1 g of KNO_3 in 100 ml of distilled water) in the cell.
Nickel sulfamate bath: Add 30 µl of 1+99 diluted sample.
Nickel sulphate and hypophosphite bath: Add 0.2-1 ml of 1+99 diluted sample
Deareate for 2 min.
Scan sample voltammogram.
Add known volumes of standard solution and scan the voltammograms.
Standard for the additions: 100 mg/l Ni^{2+}
Volume of the additions: 300 – 500 µl
Technique: DPV/a
Start potential: -700 mV
End potential finale: -1300 mV
Scanning speed: 40 mV/sec.

Analysis of Lead

Pour 10 ml of distilled water in the cell.
Add 200 µl of sample.
Deareate for 2 min.
Scan sample voltammogram.
Add known volumes of standard solution and scan the voltammograms.
Standard for the additions: 1 mg/l Pb
Volume of the additions: 100 µl
Technique: DPS/a
Deposition and start potential: -500 mV
End potential finale: -200 mV
Deposition time: 60 s
Scanning speed: 40 mV/sec.

Analysis of Cadmium

Pour 10 ml of distilled water in the cell.
Add 200 µl of sample.
Deareate for 2 min.
Scan sample voltammogram.
Add known volumes of standard solution and scan the voltammograms.
Standard for the additions: 1 mg/l Cd
Volume of the additions: 100 µl
Technique: DPS/a
Deposition and start potential: -800 mV
End potential finale: -400 mV
Deposition time: 60 s
Scanning speed: 40 mV/sec.

Analysis of Iron

Pour 10 ml of TEA / NaOH buffer (4.5 g of triethanolamine +0.8 g of NaOH in 100 ml of distilled water) in the cell.
Add 0.3 – 1 ml of sample.
Deareate for 10 min.
Scan sample voltammogram. If a shoulder appear before the maximum of the iron peak add 200 µl (or more) of 0.1 M EDTA-Na_2 and repeat the scanning..
Add known volumes of standard solution and scan the voltammograms.
Standard for the additions: 10 mg/l Fe
Volume of the additions: 100 – 500 µl
Technique: DPV/a
Start potential: -700 mV
End potential finale: -1300 mV
Scanning speed: 40 mV/sec.

Analysis of Chloride

Pour 10 ml of 0.1 M KNO_3 (1 g of KNO_3 in 100 ml of distilled water) in the cell.
Add 0.3 –0.5 ml of 1+99 diluted sample
Deareate for 5 min.
Scan sample voltammogram.
Add known volumes of standard solution and scan the voltammograms.
Standard for the additions: 10 mg/l Cl^-
Volume of the additions: 500 µl
Technique: DPV/a
Start potential: +100 mV
End potential finale: +450 mV
Scanning speed: 30 mV/sec.

Analysis of Copper

Pour 10 ml of 0.1 M $H_2C_2O_4$ - 0.2 M HCl buffer (0.9 g of $H_2C_2O_4$ or 1.26 g of $H_2C_2O_4 \cdot H_2O$, + 1.7 ml of 37% HCl in 100 ml of distilled water) in the cell.
Add 200 µl of sample.
Deareate for 2 min.
Scan sample voltammogram.
Add known volumes of standard solution and scan the voltammograms.
Standard for the additions: 1 mg/l Cu
Volume of the additions: 300 µl
Technique: DPS/a
Deposition and start potential: -400 mV
End potential finale: 0 mV
Deposition time: 60 s
Scanning speed: 40 mV/sec.

Analysis of Boric acid

Accurate rinse all the glassware in order to eliminate every acidic traces.
Pour 10 ml of distilled water in the cell, add 0.1 g of KNO_3 , 0.8 g of mannitol and 100 µl of sample
Wait 30 min.
Deareate for 3 min.
Scan sample voltammogram.
Add known volumes of standard solution and scan the voltammograms.
Standard for the additions: 1 g/l H_3BO_3
Volume of the additions: 50 µl (make not more than 2 additions)
Technique: DPV/a
Start potential: -1000 mV
End potential finale: -1800 mV
Scanning speed: 30 mV/sec.

Analysis of Saccharin

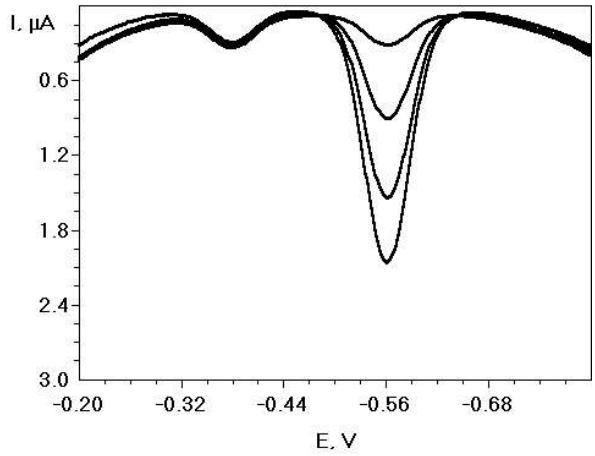
Pour 10 ml of sample in a 100 ml separatory funnel.
Add 1 ml of 37 % HCl and 5 ml of methanol.
Extract for 4 times with 10 ml of ethyl ether.
Collect all the extracts and evaporate to dryness in a rotatory evaporator.
Dry in oven at 110°C.
Add 10ml of 0.1 M HCl and dissolve the residue (treated sample solution).
Pour 10 ml of 0.1 M HCl in the cell and add 50-300 µl of the treated sample solution (above)
Deareate for 2 min.
Scan sample voltammogram.
Add known volumes of standard solution and scan the voltammograms.
Standard for the additions: 1 g/l Saccharin
Volume of the additions: 100 µl
Technique: DPV/a
Start potential: -900 mV
End potential finale: -1300 mV
Scanning speed: 30 mV/sec.

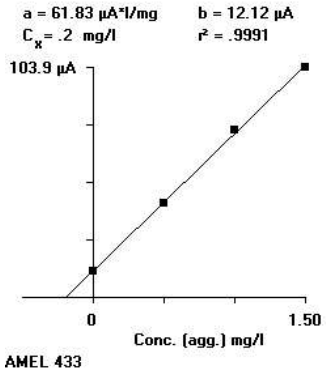
Analysis of Thiourea

Pour 10 ml of distilled water in the cell, add 50 µl of 96%H_2SO_4 and 1 – 3 ml of sample.
Deareate for 10 min.
Scan sample voltammogram.
Add known volumes of standard solution and scan the voltammograms.
Deareate for 2 min after each addition.
Standard for the additions: 10 mg/l Thiourea
Volume of the additions: 200 µl
Technique: DPV/a
Start potential: -250 mV
End potential finale: +300 mV
Scanning speed: 20 mV/sec.

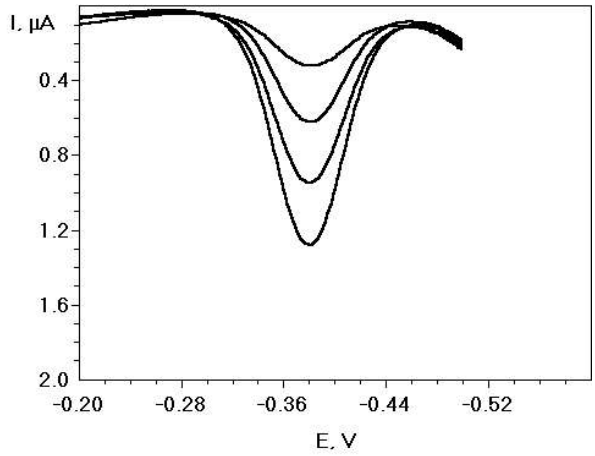
Analysis of Nickel - hypophosphite bath

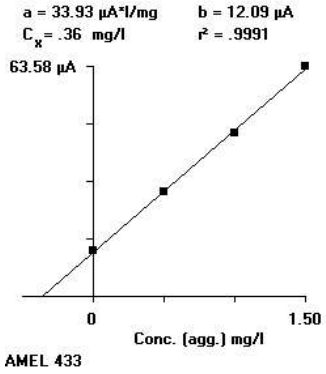
Cadmium (0.2 mg/l – dilution = 50)



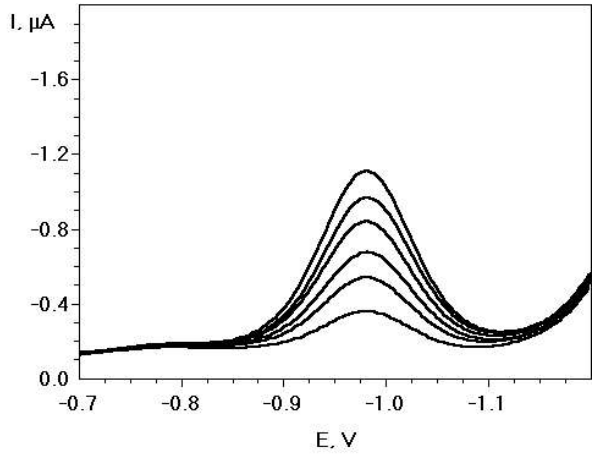


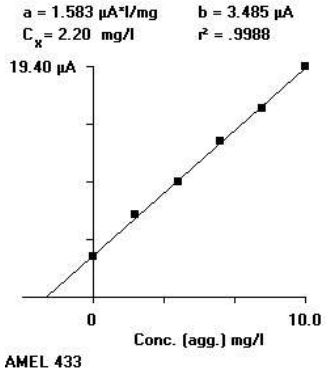
Lead (0.36 mg/l – dilution = 50)



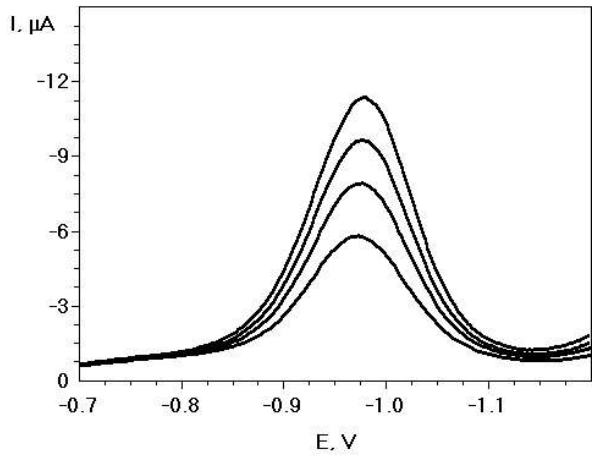


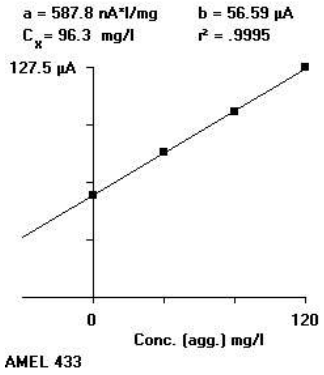
Iron (2.2 mg/l - dilution = 20)



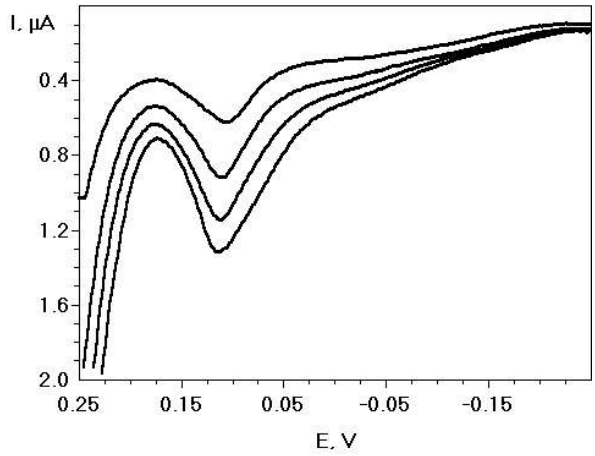


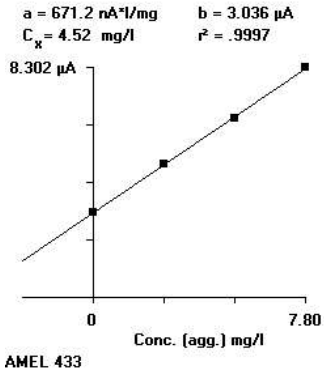
Nickel (9.6 g/l – dilution = 100)





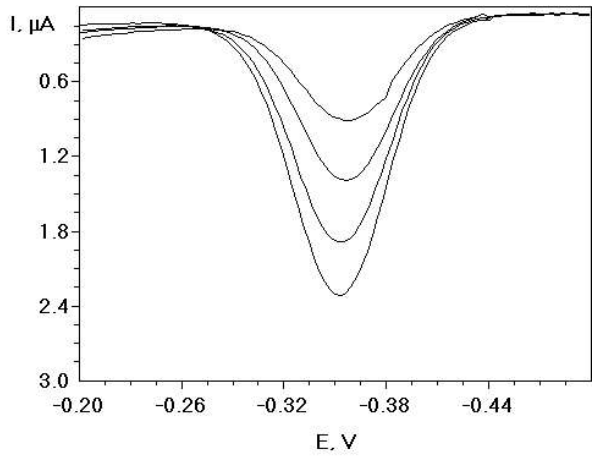
Thiourea (4.5 mg/l – dilution= 1)

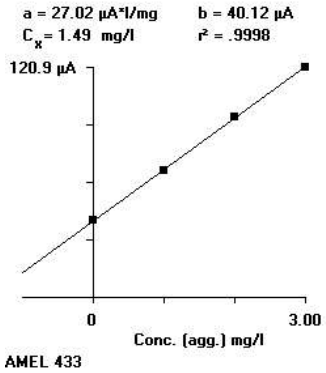




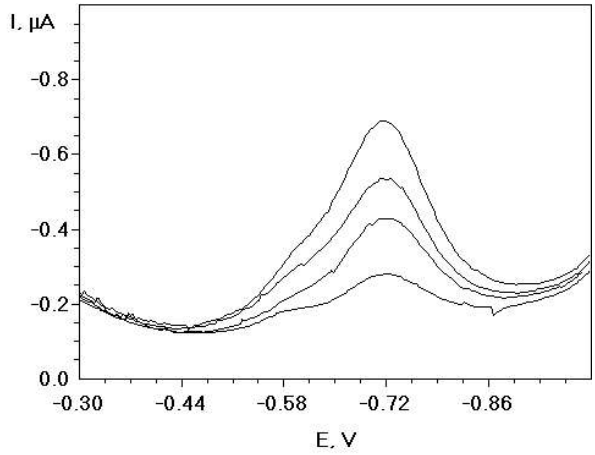
Analysis of Nickel - sulfamate bath

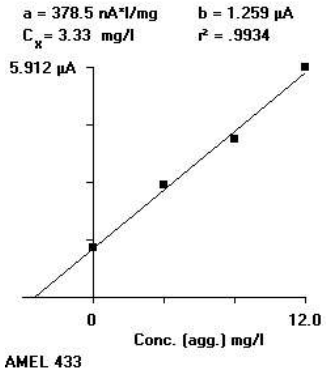
Lead (1.5 mg/l – dilution = 50)



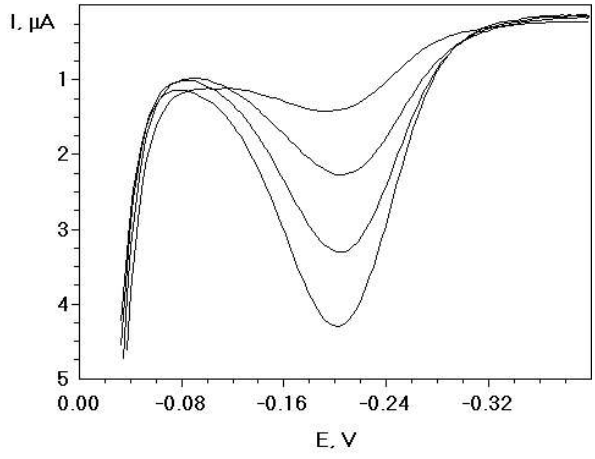


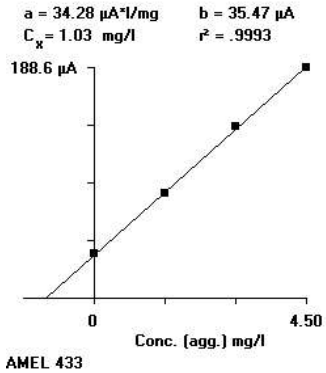
Iron (3.3 mg/l – dilution = 10)



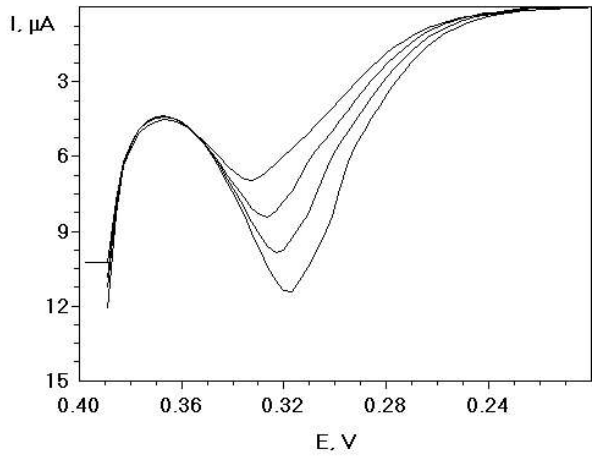


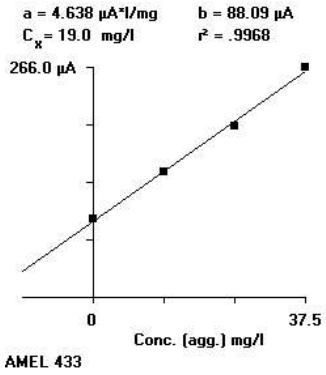
Copper (1.0 mg/l – dilution =50)



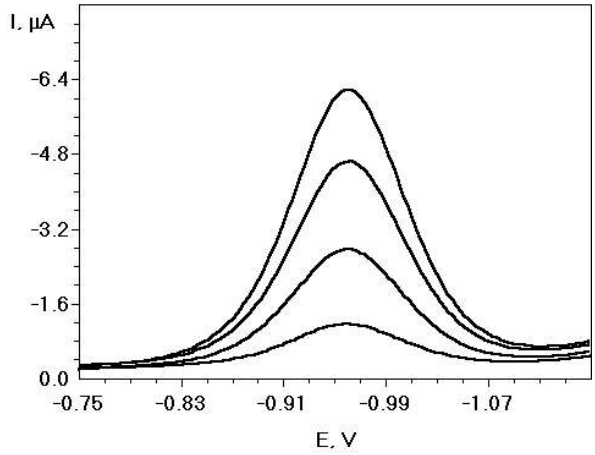


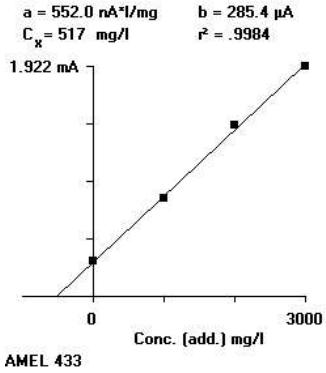
Chloride (1.9 mg/l – dilution = 100)



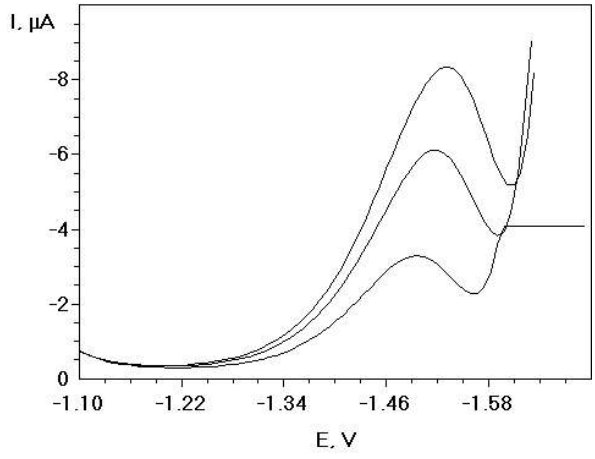


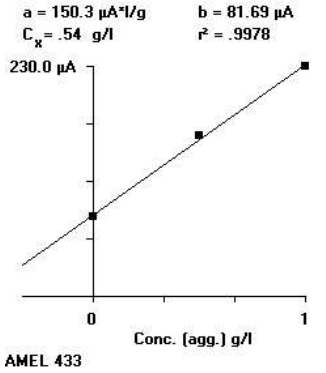
Nickel (52 g/l – dilution = 100)





Boric acid (0.54 g/l – dilution =1)





Saccharin (0.65 g/l – dilution 3.3)