# VA Application Note No. V - 151

| **Title:** | **Antimony(III) and total antimony in electroless nickel bath** |
|---|---|


| ***Summary:*** | The concentration of Sb(III) and Sb(total) in an electroless nickel bath is determined by anodic stripping voltammetry (ASV). In c(HCl) = 0.6 mol/L only Sb(III) shows a signal. In w(HCl) = 10% the Sb(total) content is determined. |
|---|---|


| ***Sample:*** | Electroless Ni bath |
|---|---|
| ***Sample preparation:*** | None |

## Analysis of Sb(III)

| | | |
|---|---|---|
| ***Electrolyte for Sb(III)*** | c(HCl) = 0.6 mol/L | |
| ***Measuring solution*** | 15 mL electrolyte for Sb(III)<br>+ 50 µL Ni plating bath | |
| ***Working electrode (WE)*** | **MME** (Multi Mode Electrode) | 6.1246.020 |
| ***Auxiliary electrode (AE)*** | **Pt** | 6.0343.000 |
| ***Reference electrode (RE)*** | Reference system: Ag/AgCl/KCl (3 mol/L)<br>Intermediate electrolyte: c(KCl) = 3 mol/L | 6.0728.020<br>6.1245.010 |

***Parameters***

| | |
|---|---|
| Working electrode | HMDE |
| Stirrer speed | 2000 rpm |
| Mode | DP |
| Purge time | 300 s |
| Deposition potential | -0.45 V |
| Deposition time | 30 s |
| Equilibration time | 10 s |
| Pulse amplitude | 0.02 V |
| Start potential | -0.3 V |
| End potential | 0 V |
| Voltage step | 0.004 V |
| Voltage step time | 0.3 s |
| Sweep rate | 0.013 V/s |
| Peak potential Sb(III) | -0.12 V |

## Analysis of Sb(total)

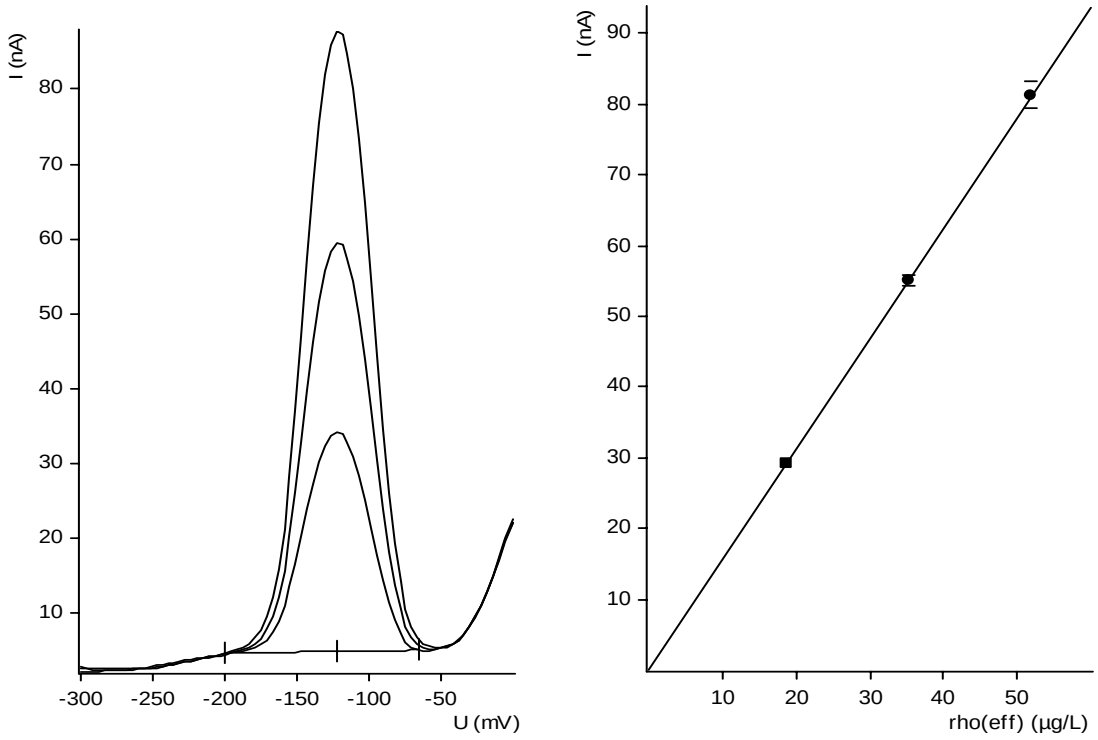
| | | |
|---|---|---|
| ***Electrolyte for Sb(total)*** | w(HCl) = 10% | |
| ***Measuring solution*** | 15 mL electrolyte for Sb(total)<br>+ 20 µL Ni plating bath | |
| ***Working electrode (WE)*** | **MME** (Multi Mode Electrode) | 6.1246.020 |
| ***Auxiliary electrode (AE)*** | **Pt** | 6.0343.000 |
| ***Reference electrode (RE)*** | Reference system: Ag/AgCl/KCl (3 mol/L)<br>Outer system: c(KCl) = 3 mol/L | 6.0728.020<br>6.1245.010 |

***Parameters***

| | |
|---|---|
| Working electrode | HMDE |
| Stirrer speed | 2000 rpm |
| Mode | DP |
| Purge time | 300 s |
| Deposition potential | -0.45 V |
| Deposition time | 30 s |
| Equilibration time | 10 s |
| Pulse amplitude | 0.02 V |
| Start potential | -0.3 V |
| End potential | -0.1 V |
| Voltage step | 0.004 V |
| Voltage step time | 0.3 s |
| Sweep rate | 0.013 V/s |
| Peak potential Sb(total) | -0.19 V |

| **Results:** | Sb(III) | Sb(total) |
|---|---|---|
| | 5.6 mg/L | 47.3 mg/L |

## Determination of Sb(III)




## Determination of Sb(total)