VA Application Note No. V-77

Title:	Nickel and cobalt in zinc plant electrolytes (concentrated $ZnSO_4$ solutions)

Summary:	Nickel can be determined in concentrated zinc solutions by adsorptive stripping voltammetry (AdSV) at the HMDE using ammonia buffer as supporting electrolyte and dimethylglyoxime (DMG) as complexing agent. The determination of cobalt does not work under these conditions as the very high Zn^{2+} concentration interferes with the Co signal. Therefore an alternative complexing agent has to be used: α-benzil dioxime in ammonia buffer under addition of sodium nitrite.

Sample:	Zinc plant electrolyte
Sample preparation:	none

Determination of nickel

Electrolyte	Ammonia buffer: c(HCl) = 1 mol/L + c(NH_3) = 3 mol/L DMG solution: c(dimethylglyoxime disodium salt) = 0.1 mol/L in water Citrate solution: c(Na_3 citrate * 2 H_2O) = 1 mol/L	
Measuring solution	0.5 mL sample + 5 mL ultrapure water + 5 mL citrate solution + 1 mL ammonia buffer + 100 µL DMG solution	
Auxiliary electrode (AE)	Pt	
Reference electrode (RE)	Ag/AgCl/KCl (3 mol/L)	
Parameters	Working electrode	HMDE
	Stirrer speed	2000 rpm
	Mode	DP
	Purge time	300 s
	Deposition potential	–700 mV
	Deposition time	60 s
	Equilibration time	10 s
	Pulse amplitude	50 mV

	Start potential	–700 mV
	End potential	–1100 mV
	Voltage step	4 mV
	Voltage step time	0.3 s
	Sweep rate	13.3 mV/s
	Peak potential Ni	–950 mV

Determination of cobalt

Electrolyte

α-Benzil dioxime solution:
c(α-benzil dioxime) = 1 mmol/L

Weigh the α-benzil dioxime into a 100 mL volumetric flask and add 20 mL ethanol. Add c(NaOH) = 1 mol/L until the α-benzil dioxime has completely dissolved, then add 10 ml ultrapure water and make up to 100 mL with ethanol.

Supporting electrolyte:
c($NaNO_2$) = 0.5 mol/L
+ c(NH_3) = 0.55 mol/L
+ 6.5 mL/500 mL α-benzil dioxime solution,
adjust pH to 9.2 ± 0.1

Measuring solution

10 mL supporting electrolyte
+ 0.1 mL diluted sample (1 : 10 in water)

Auxiliary electrode (AE)

Pt

Reference electrode (RE)

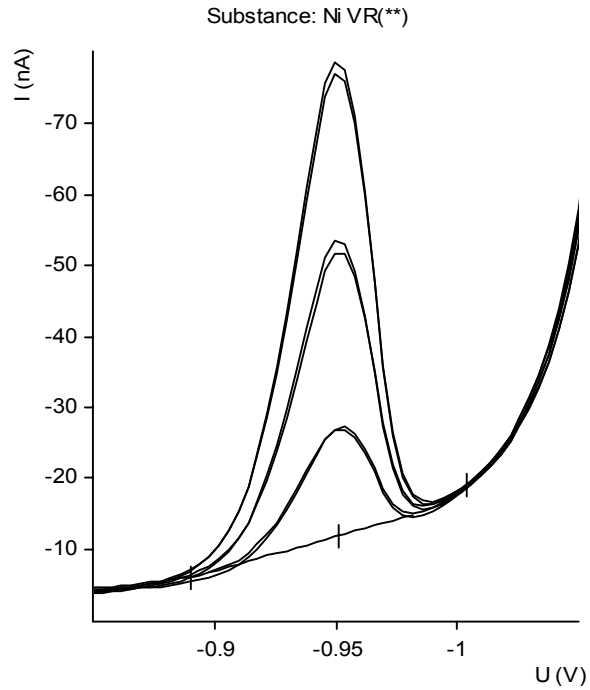
Ag/AgCl/KCl (3 mol/L)

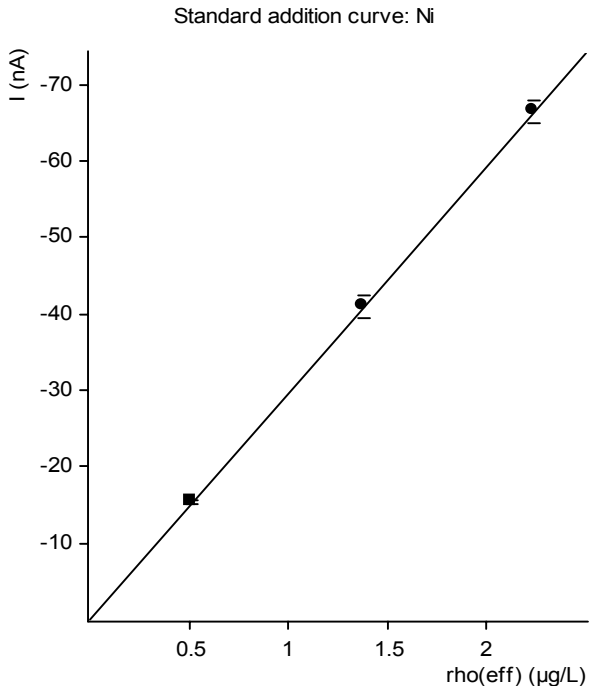
Parameters

Working electrode	HMDE
Stirrer speed	2000 rpm
Mode	DP
Purge time	300 s
Deposition potential	–800 mV
Deposition time	20 s
Equilibration time	10 s
Pulse amplitude	50 mV
Start potential	–800 mV
End potential	–1125 mV
Voltage step	4 mV
Voltage step time	0.1 s
Sweep rate	40 mV/s
Peak potential Co	–960 mV

Results:	Ni	Co
	12 µg/L	105 µg/L

Determination of Ni





Determination of Co