Determination of Nickel in Plating Bathes (potentiometric)

Description

The determination of Nickel in plating bathes is done by titration with Ethylenediaminetetraacetic acid (EDTA, Na-salt). A copper electrode is used for the detection of the equivalence point.
Since the Cu electrode can only detect Cu ions, a small amount of Cu-EDTA complex must be added. The nickel from the sample displaces the Cu from the Cu-EDTA complex, so that free Cu ions are also present - when all the Ni from the sample is complexed, the Cu is complexed again, and the EQ can be detected.
Other complexable metal ions such as calcium, magnesium, zinc or copper are titrated with.
For low Ni contents a 0.05 mol/L EDTA solution can be used instead of the 0.1 mol/L.

Instruments

Titrator	TL 5000, TL 7000, TL 7750, TL 7800
Electrode	Cu 1100 PLH
Cable	L 1 A
Reference electrode	B 2920+
cable	L 1 N
Stirrer	Magnetic stirrer TM 235
Lab accessory	Glass beaker 150 ml
	Magnetic stirrer bar 30 mm

Reagents

1	Na_2EDTA 0.1 mol/l
2	Ammonia solution 25%
3	Ammonium chloride
4	Copper-EDTA solution 0.1 mol/l ($Cu(NH_4)_2$-EDTA)
5	Electrolyte solution L3004
6	Distilled Water
All reagents should be of analytical grade or better.	

Titration procedure

Reagents

EDTA solution 0.1 mol/L
EDTA solution 0.1 mol/L is available as ready-to-use solution.
The titer determination of the EDTA solution is done as described in the application note "Titer determination of EDTA".

Buffer solution pH 10
54.0 g of Ammonium chloride are dissolved in a little water, 350 ml of Ammonia solution 25% are added and made up to 1.0 liter with dist. Water.

Cleaning of the electrode

The electrodes are cleaned with distilled water. The Cu 1100 is stored clean and dry, for the storage of the reference electrode electrolyte solution L300 is used.

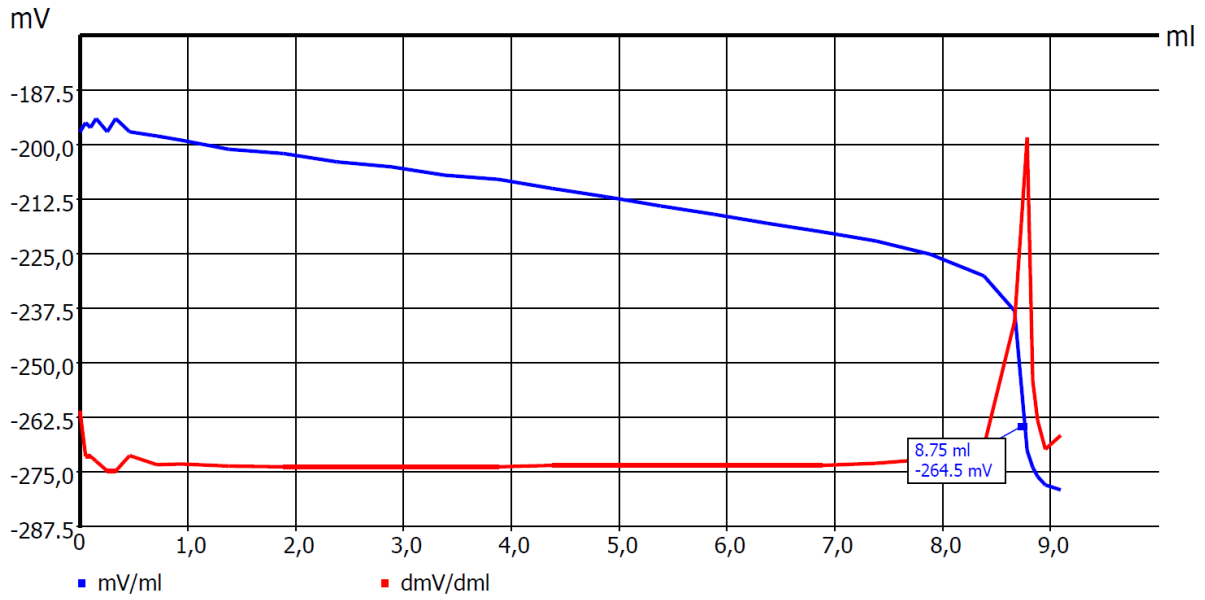
Sample preparation

The sample is pipetted into a 150 ml beaker and made up to 60 – 80ml. 10 ml of buffer solution pH 10 and 1 ml of Cu-EDTA 0.1 mol/l are added. The pH of this solution should be approximately pH 10. Then it is titrated with EDTA 0.1 mol/L to an Equivalence point. The consumption should be about 5 - 15 ml.

The required sample amount can be estimated according to this rule of thumb:

$$V(mL) = \frac{590 * Titer\ [\frac{mol}{L}]}{expected\ Ni - content\ [g/L]}$$

Titration parameter



Default method	---		
Method type	Automatic titration		
Modus	Dynamic		
Measured value	mV		
Measuring speed / drift	User - defined	Minimum holding time	5 s
		Maximum holding time	12 s
		Measuring time	4 s
		Drift	3 mV/min
Initial waiting time	5 s		
Dynamic	Flat	Max step size	0.5 ml
		Slope max ml	10
		Min. step size	0.05 ml
		Slope min. ml	120
Damping	none	Titration direction	decrease
Pretitration	off	Delay time	0 s
End value	off		
EQ	On (1)	Slope value	120
Max. titration volume	20 ml		
Dosing speed	100%	Filling speed	30 s

Calculation in g/L:

$$Nickel\ [g/l] = \frac{(EQ1 - B) * T * M * F1}{V * F2}$$

B	0	Blank value
EQ1		Consumption of titrant at first Equivalence point
T	WA	Actual concentration of the titrant
M	58,69	Molecular mass of Nickel
V	man	sample volume [ml]
F1	1	Conversion factor
F2	1	Conversion factor

Calculation in mol/L:

$$Result\ [mol/l] = \frac{(EQ1 - B) * T * M * F1}{V * F2}$$

B	0	Blank value
EQ1		Consumption of titrant at first Equivalence point
T	WA	Actual concentration of the titrant
M	1	
V	man	sample volume [ml]
F1	1	Conversion factor
F2	1	Conversion factor

Any questions? Please contact the application team:

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