Application Bulletin

Of interest to:	Plating industry, semiconductor industry, automotive industry, aeronautic industry, electroless nickel plating, hypophosphite, alkalinity	P 9, 10

Determination of nickel, hypophosphite and alkalinity in electroless nickel plating baths

Summary

Electroless nickel layers are normally nickel-phosphorus alloys. For the reductive chemical deposition of nickel with phosphorus inclusions, sodium-hypophosphite is used as the reducing agent (reductant).

To apply even nickel layers to the workpieces, the content of nickel, reductant and the alkalinity have to be determined periodically.

For the determination of the ingredients an automated ProcessLab system is used. All liquid handling steps such as taking sample aliquots, dosing of reagents, titration and cleaning are performed by pumps and burets controlled by ProcessLab.

A sample aliquot is transferred either to the vessel for alkalinity and nickel analysis or to the vessel for reductant content determination.

Features/general information

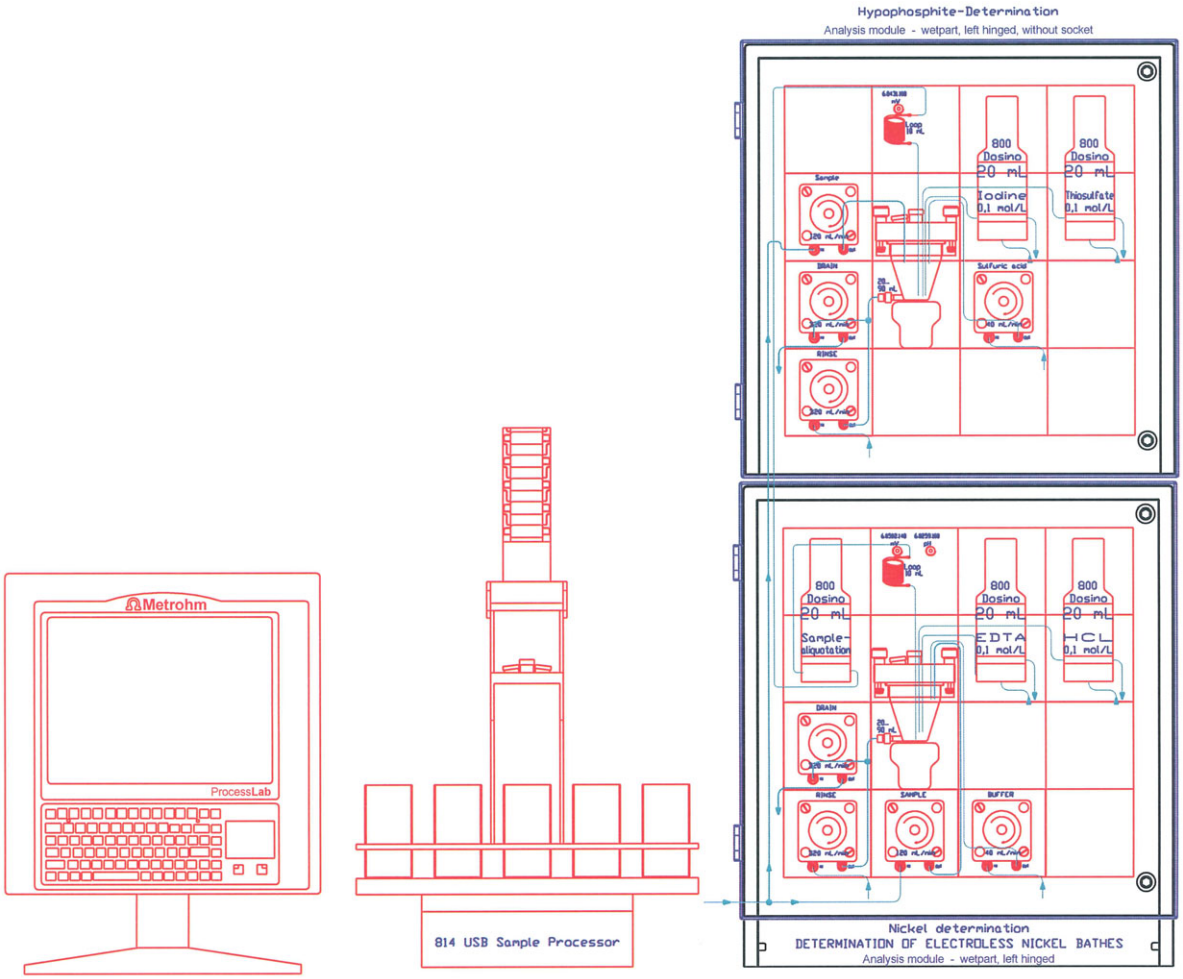
- Alkalinity, nickel and hypophosphite determination in one system
- Barcode reader offers automatic method selection by using predefined bar-codes
- Optional liquid level sensors monitor the reagent level and call for manual intervention when required
- Additional terminals for the I/O controller allow to transmit or receive external signals, for example a result that is sent to a remote PCS (Process Control System). An incoming signal can be a density value from a density meter.

This system is very flexible and can be adapted very easily to any specific needs. If necessary an additional reagent cabinet is available and ProcessLab can be placed on top of it. There is sufficient space for all reagents, which makes ProcessLab even more practical.

Accessories used (only the most important parts are listed)

- 1 x 2.875.0010; 875 ProcessLab Base Unit L, 1 Metrohm Dosing & Measuring Controller (incl. IPC, I/O controller and TFT/Keyboard terminal)
- 1 x 2.875.0130; ProcessLab Extension module L, 1 Metrohm Dosing & Measuring Controller
- 3 x 6.7201.000; Measuring amplifier
- 5 x 2.800.0010; 800 Dosino
- 5 x 6.3032.220; Buret 20 mL
- 1 x 6.7206.040; Dosino sample loop 10 mL, variable
- 2 x 6.7205.010; Peristaltic pump, 40 mL/min
- 2 x 6.7205.020; Peristaltic pump, 120 mL/min
- 4 x 6.7205.030; Peristaltic pump, 320 mL/min
- 1 x 2.814.0030; USB Sample processor with 1 tower
- 1 x 6.0431.100; Pt Titrode
- 1 x 6.0502.140; Cu ISE
- 1 x 6.0259.100; LL Unitrode
- Different input and output terminals, e.g. digital or analog output
- Barcode reader
- Reagent containers if needed (5, 10 and 20 L), incl. level switch

Wet part setup & system overview



Reagents

- Iodine titrant, c(I_2) = 0.1 mol/L
- Thiosulfate titrant, c($Na_2S_2O_3$) = 0.1 mol/L
- EDTA titrant c(EDTA) = 0.1 mol/L
- Hydrochloric acid titrant, c(HCl) = 0.1 mol/L
- Buffer reagents depending on the analysis, e.g. sulfuric acid and buffer pH=10
- Demineralized water

Calibration and storage of the electrodes

- The electrodes need no calibration as only inflection point titrations are carried out.
- The electrodes should be stored in demin. water, for longer times store the pH electrode and Pt Titrode in KCl 3 mol/L and the Cu ISE in a clean and dry place.

Analysis

Performing the analysis

The sample is filled into a barcode-labelled beaker and put onto the sample processor. After starting the ***tiamo*** method the barcode is scanned and the appropriate analysis tracks are selected (***tiamo*** identifies the predefined barcode string).

With this method no sample table and no manual sample information is used and urgent samples can be inserted at any time.

Alkalinity determination – titration with HCl

The sample aliquot is filled up with demin. water and titrated to the first inflection point with hydrochloric acid standard solution against a combined pH-glass electrode. As soon as the first inflection point is reached, ***tiamo*** calculates the results and, depending on the barcode, either triggers cleaning of the vessel or continues with the nickel determination.

Nickel determination – titration with EDTA

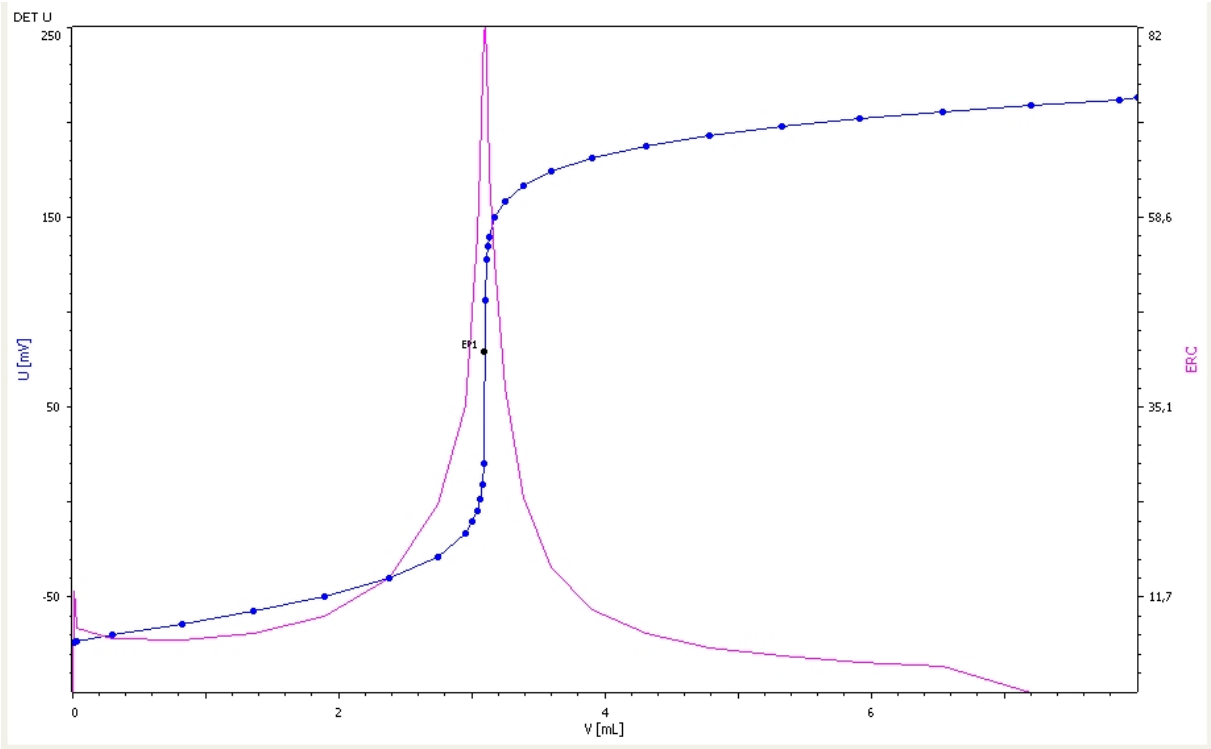
Addition of a defined amount of buffer pH = 10 and titration with EDTA standard solution to one inflection point against a combined Cu-sensitive electrode. When the titration is finished the vessel is cleaned; ***tiamo*** calculates the result and creates the database entry.

Hypophosphite determination – titration with iodine and thiosulfate

A defined amount of iodine solution is added to the sample aliquot and acidified with sulfuric acid. After the wait time the iodine that has not reacted with hypophosphite is titrated to one inflection point with thiosulfate standard solution against a Pt Titrode. When the titration is finished the vessel is cleaned; ***tiamo*** calculates the result and creates the database entry.

Practical example

Back-titration of iodine using thiosulfate



Literature

Gros, Prof. Dr. Leo, Bruttel, P., von Kloeden, M. (2005) Practical titration, Metrohm Monograph 8.029.5003

Haider, Dr. C., Electrodes in Potentiometry, Metrohm Monograph 8.015.5003