Application Bulletin

Of interest to:	General analytical chemistry, metals, electroplating, automotive, phosphatizing	P 1, 10

Monitoring parameters in a phosphatizing process (pH, conductivity, acidity, alkalinity, fluoride and zinc)

Summary

The quality of a phosphatized metal surface directly depends on the composition of the phosphatizing baths. Because the constant loss of bath reagents or contamination of the rinsing baths it is very important to constantly monitor these agents. Keeping the consumption of phosphatizing agents as low as possible can optimize costs.

In a modern factory you find many different types of baths. It is very difficult to keep an eye on every single parameter. ProcessLab will monitor these by itself and, if desired, transmit the data to any suitable process control system (PCS). Alarm criteria can be set as well as external signals like a flashing alarm lamp.

ProcessLab can, with the help of barcodes, automatically recognize the sample and the parameter which have to be analyzed. Place any sample on ProcessLab, register it and hit the “Start” button. All you get is the result at the desired place like e.g. a report or a simple PDF. Custom-designed for the particular needs of the process.

The described ProcessLab at-line analysis system controls, records and documents the important analytical parameters of the entire phosphatizing process. The combination of the analytical methods involved as well as the intuitive handling via the well-arranged user interface allow for complete process control.

Features/general information

- All 6 parameters in one system and monitored on demand
- OneButton analysis
- Very easy to operate for semi-skilled staff
- One sample cycle includes preparation of Dosino, rinsing, sampling and measuring
- Barcode reader offers automatic method selection
- Optional liquid level sensors monitor the reagent level and advice ProcessLab to wait for manual intervention if necessary
- Additional terminals for the I/O controller allow an external communication like transmission of a result to a PCS. An “out of limit” alarm can be set and any external device will be switched on.

This system is very flexible and can be adapted very easily to any specific needs. If necessary an additional reagent cabinet is available and placed directly under the ProcessLab module. It has sufficient space for all reagents and makes ProcessLab even more comfortable.

Used Accessories (some are optional)

- 1 x 2.875.0030; 875 ProcessLab Base Unit L, 2 Metrohm Dosing & Measuring Controller (incl. IPC, I/O controller and TFT/Keyboard terminal)
- 1 x 2.875.0140; 875 Extension module R, 1 Metrohm Dosing & Measuring Controller
- 4 x 6.7201.000; Measuring amplifier
- 1 x 2.712.0010; 712 Conductometer
- 7 x 2.800.0010; 800 Dosino
- 5 x 6.3032.220; Buret 20 mL
- 2 x 6.3032.250; Buret 50 mL
- 13 x 6.7205.0X0; Peristaltic pump
- 1 x 6.0901.260; Conductivity cell
- 2 x 6.0259.100; Unitrode
- 1 x 6.0351.100; Pt ring electrode
- 1 x 6.0502.140; Cu ISE
- 1 x 6.0502.150; F ISE
- 1 x 2.814.0030; 814 Robotic USB Sample processor without pumps/valves
- 1 x 6.2041.810; Sample rack, single row
- 4 x 6.7202.100; Digital input 4 DI 24V DC
- 5 x 6.7202.200; Digital output 4 DO 24V DC
- 5 x 6.7202.500; Relay output 2 DO Relay
- 6.7207.0X0; Reagent containers (2.5, 5, 10 and 20L available), incl. level sensor

Wet part layout

Reagents

- Titrant c(HCl) = 0.1 mol/L
- Titrant c(NaOH) = 0.1 mol/L
- Titrant c(EDTA) = 0.1 mol/L
- F standards
- TISAB IV
- Buffer pH=4 and 7 for calibration of the electrodes
- Conductivity standards
- Demineralized water

Calibration and storage of the sensors

- The electrodes need to be calibrated on a regular basis, e.g. once a week
- When not used the Unitrode is stored in 3mol/L KCl (250 mL; 6.2308.020)
- Conductivity cells need a special and regular maintenance

Analysis

Conductivity

The conductivity is measured first using a conductivity cell and a conductometer on the sample changer. It is important that the sample is measured directly after placing it because conductivity changes quickly due to different factors.

pH

Is measured as well on the sample changer after conductivity measurement. pH measurement is easily and quickly done.

Free and total alkalinity

The sample is titrated with hydrochloric acid and the values are obtained from the first and second inflection point.

Free and total acid

These values are determined by a normal titration with NaOH and the endpoints are used for the calculations.

Zinc

Zinc is measured using a complexometric titration method with EDTA and the Cu ISE. Make sure there are no other metals that form an EDTA complex.

Fluoride

The fluoride content is determined by using a fluoride ISE and a direct potentiometric measurement. The solution is spiked with a known concentration of fluoride. The resulting potential difference is used to calculate the initial concentration of fluoride. This is the only measurement that is done in the left titration vessel.

Summary

All analysis can be done using only two titration cells, these are placed in 2 of the ProcessLab modules. All necessary pumps and Dosinos are included and autonomously controlled by *tiamo*. Conductivity and initial pH measurement are performed directly on the sample processor.

Acid-base determinations and zinc titration are all accomplished in the right titration cell. Only the direct potentiometric fluoride measurement is performed in the left vessel.

Literature

Portala, F. Müller, F. Feige, M. Kirner. G. (2006) Poster: "Monitoring of a complete process line for the phosphatizing of metal surfaces using an at-line analysis system", PDF document, www.metrohm-processlab.com