XRF inline solution analysis of zinc-nickel concentration of electroplating baths

Zinc-nickel coatings are widely used because of their excellent corrosion-protective characteristics. It is important for the quality of these and other metallic coatings that the composition of the electroplating bath is within a narrow tolerance window. This is the only way to achieve the required coating properties. For efficient bath monitoring and control, inline measurement by X-ray fluorescence analysis is a suitable method.

Status quo and challenges

Electroplating baths are subject to process fluctuations which can have a considerable influence on the deposition behavior and the resulting coating thickness. Continuous monitoring of the solutions is therefore essential. For this, the zinc-nickel (ZnNi) concentrations of the baths are regularly checked manually in the company laboratory, for example using wet chemistry, AAS or ICP-OES methods. In general, such measurements take place 1 to 2 times a day. The disadvantage of these chemical procedures is that they are time-consuming and cost-intensive. Due to the time offset between sampling and testing, real-time bath control is also not possible.
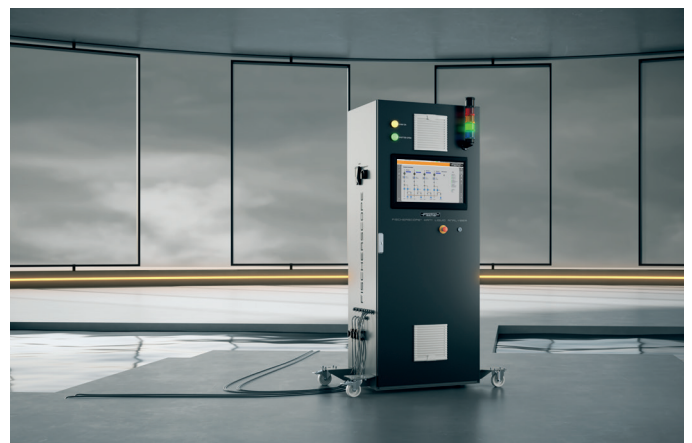

Figure 1: The solution for your solutions: The FISCHERSCOPE® XAN® LIQUID ANALYZER

A more frequent bath control is desirable for process-related reasons. In order to be able to run the deposition process safely and without much waste, at least four zinc-nickel bath analyses per hour should have been carried out. These frequent measurements can no longer be reasonably carried out manually, so automating the measurement would lead to more efficiency in the process.

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In theory, the chemical measurement methods listed above can be automated. Due to the high salt load and aggressiveness of the ZnNi baths, the X-ray fluorescence measurement method is particularly suitable for automation, as it does not require any further sample preparation compared to the other methods.

Automated inline quality control for electrolyte solutions

The FISCHERSCOPE® XAN® LIQUID ANALYZER in combination with the powerful and proven WinFTM® software combines all requirements: The fully automated inline XRF measuring instrument continuously delivers measurement results and analyzes the metal concentration of up to four electroplating baths with high precision. The calibration is also fully automatic and the intuitive and large touch display ensures easy operation. The instrument is not only suitable for the control of zinc-nickel baths, but also for the monitoring of other galvanic baths such as zinc, nickel, gold, chromium, rhodium and palladium.

The high-tech measuring cell is unique: the flow cell is suitable for the temperatures, pH values, salt loads, solvents and particle loads typical for the process and, due to the design, no change of the measuring cell is necessary. In combination with intelligent material selection and automatic rinsing and monitoring processes, a maximum tested uptime of more than one year is guaranteed, depending on rinsing and cleaning cycles and the composition of the coating baths, among other factors. The design of the measuring device with four separate inline measuring cells as well as inlets and outlets - a separate measuring cell for each bath to be monitored - simplifies bath inlet, saves valves and prevents contamination.

Functionality

The FISCHERSCOPE® XAN® LIQUID ANALYZER is connected to the electroplating bath either directly via a line or via a ring line. The solution to be analyzed is delivered to the flow cell in the device, in which the metal content is determined fully automatically by X-ray fluorescence measurement.

The measured values are displayed on a screen (HMI) or exported to the higher-level control system. The special feature: The measured data can be displayed simultaneously on additional monitors directly at the electroplating bath. The plant operator can therefore monitor the current measured values and react quickly in the event of deviations, even if the measuring device is located in a separate room.
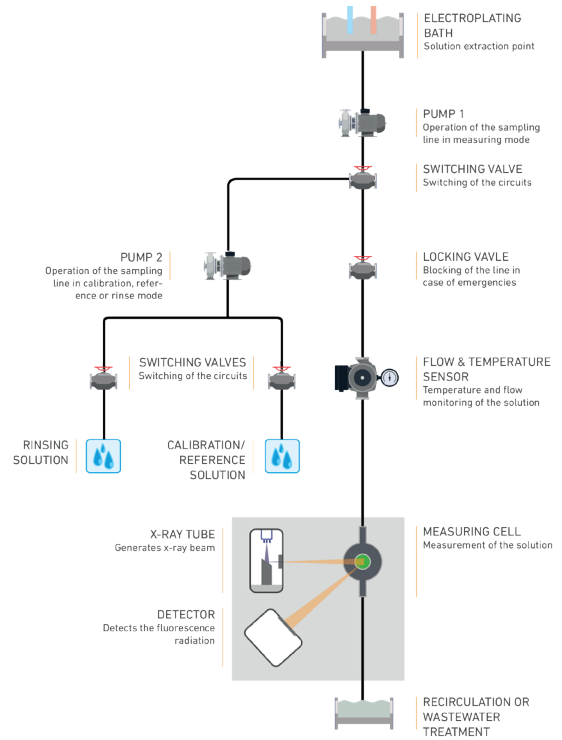

Figure 2: Functionality of the FISCHERSCOPE® XAN® LIQUID ANALYZER

Market leading measurement performance

As an example, measurements were carried out on various typical ZnNi baths. The typical ZnNi bath is composed of 5 - 15 g/l zinc and 0.5 - 2 g/l nickel.

In a first step, the comparability of the XRF measurement with a classical wet chemical analysis (titration) was compared. For this purpose, the concentration of zinc and nickel was measured over a measurement time of 10 seconds each.
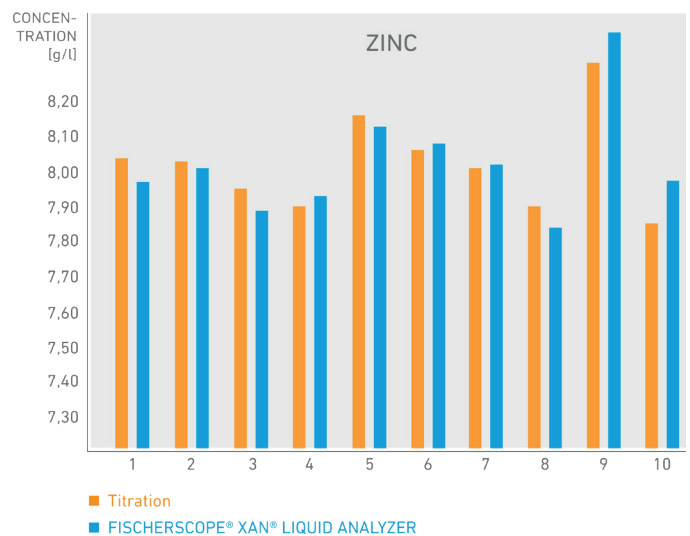

Figure 3: Comparative measurements of zinc by XRF and titration

NICKEL

CONCEN-TRATION [g/l]

■ Titration
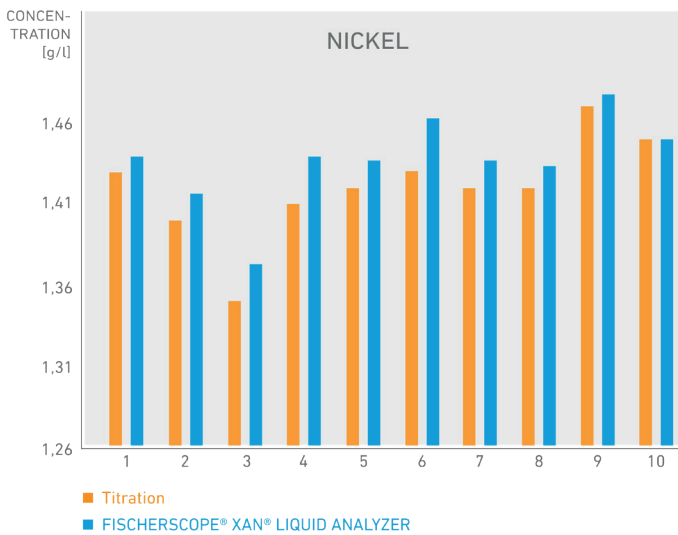■ FISCHERSCOPE® XAN® LIQUID ANALYZER

Figure 4: Comparative measurements of nickel by XRF and titration

As can be seen from Figures 3 and 4, the determination of metal concentration by XRF measurement provides very good comparability to titration.

In another measurement, the long-term measurement behavior over a period of 48 h was observed on a typical ZnNi bath.

ZINK-CONCENTRATION

CONCEN-TRATION [g/l]

■ Zn [g/l]

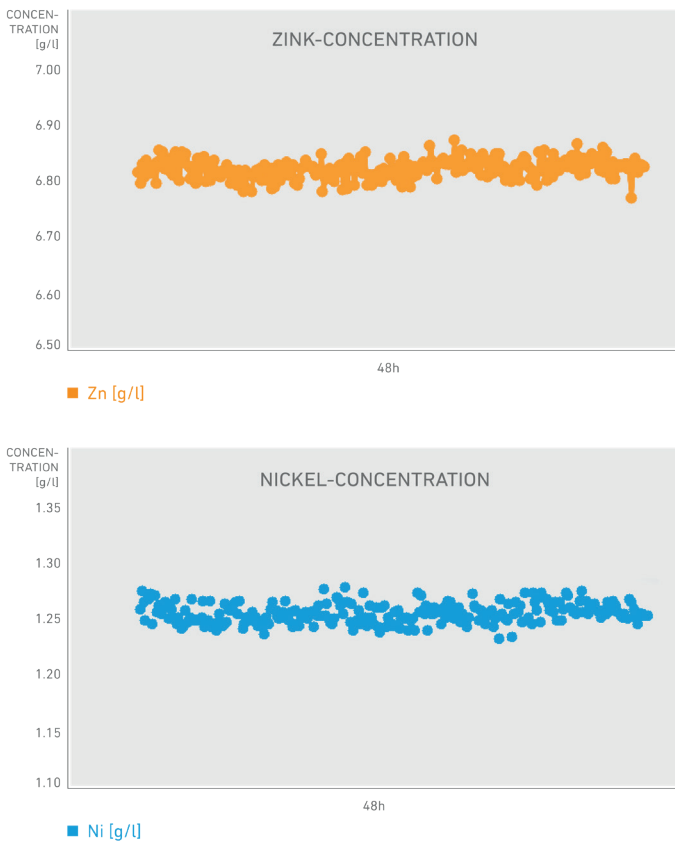NICKEL-CONCENTRATION

CONCEN-TRATION [g/l]

■ Ni [g/l]

Figure 5: Results of the long-term measurement of zinc and nickel concentrations over 48 hours

The FISCHERSCOPE® XAN® LIQUID ANALYZER shows a very good repeatability in measurements. At the measured value of 6.82 g/l zinc and 10 s measuring time, the coefficient of variation (COV) is only 0.26 %. At 1.26 g/l nickel and also 10 s measuring time, the COV is 0.71 %.

	Zink	Nickel
Average value [g / l]	6,82	1,26
Standard deviation [g / l]	0,018	0,009
Coefficient of variation COV [%]	0,26	0,71

Figure 6: Measurement results

Conclusion:
For efficient analysis of metal concentrations in electroplating baths, the XRF inline instrument FISCHERSCOPE® XAN® LIQUID ANALYZER is the perfect answer. By analyzing up to 4 electroplating baths in a row and in real time, it eliminates the need to change measuring cells, time-consuming sampling, and information gaps. The instrument measures your electroplating bath automatically and with the highest precision. It ensures reproducible measurement results that you can rely on. Without loss of time, the bath control can be adapted to the results of the measurements. This makes your solution analysis more precise, faster, more economical and essentially maintenance-free.

For further information, please contact our Fischer experts: sales@helmut-fischer.com