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Galvanic baths for the treatment of industrial fasteners

# XRF inline solution analysis of the zinc-nickel concentration of galvanic baths

Zinc-nickel coatings are widely used due to their excellent anti-corrosion properties. It is crucial for the quality of these and other metallic coatings that the composition of the galvanic bath remains within a narrow tolerance range. This is the only way to achieve the desired coating properties. Inline measurement using X-ray fluorescence analysis is ideal for efficient bath monitoring and control.

Electroplating baths are subject to process-related fluctuations, which can have a considerable influence on the deposition behaviour and the resulting coating thickness. In order to better control the deposition process and avoid excessive waste, the bath solutions must be regularly monitored by analysing the solution as often as possible. In the case of a galvanic bath with zinc-nickel solution, at least four zinc-nickel bath analyses per hour are desirable. Up to now, the metal concentration of the bath has so far been checked manually in the company laboratory, for example using wet chemistry, atomic absorption spectrometry (AAS) or inductively coupled plasma optical emission spectrometry (ICP-OES).

## X-ray fluorescence as a key technology for bath analysis

However, as these manual measurement methods are very time-consuming and cost-intensive, bath checks are usually only carried out once or twice a day. This means that the desired frequency cannot be achieved. Due to the time lag between sampling and testing, real-time bath control is also not possible.

Automating the measurement not only makes the inspection process much more efficient in many respects, but also ensures consistent product quality. In principle, the chemical analysis processes can be automated. However, due to the high salt load and aggressiveness of the zinc-nickel baths, the X-ray fluorescence measuring method is the preferred method for automation, as it does not require any further sample preparation compared to the other methods.

### Automated inline quality control for electrolyte solutions

The Helmut Fischer Group, the market-leading measurement technology specialist for coating thickness measurement and material analysis, has developed a solution to these challenges. In combination with the powerful and proven software WinFTM<sup>®</sup>, the FISCHERSCOPE<sup>®</sup> XAN<sup>®</sup> LIQUID ANALYZER fulfils all requirements for the inline solution analysis of electroplating baths.



© Helmut Fischer GmbH Institute for Electronics and Measurement Technology The solution for your solutions – The FISCHERSCOPE® XAN® LIQUID ANALYZER

The fully automated inline XRF measuring device analyses the metal concentration of up to four electroplating baths continuously and with high precision. The practical touch display is used both to display the measurement results and to control the entire measuring system centrally. Calibration is also fully automatic. The FISCHERSCOPE<sup>®</sup> XAN<sup>®</sup> LIQUID

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ANALYZER is not only suitable for monitoring zinc-nickel baths, but also for monitoring other galvanic baths made of zinc, nickel, gold, chromium, rhodium or palladium.

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### Low maintenance due to innovative measuring cell

The unique key component of the inline measuring device is the measuring cell. This is a robust flow cell that has been specially developed in-house for the temperatures, pH values, salt loads, solvents and particle loads typical of the process. Consisting of particularly hard-wearing materials, the measuring cell proves to be especially durable. In combination with automatic calibration, rinsing and cleaning processes, the measuring cell achieves an excellent service life of up to one year<sup>1</sup>, depending on the frequency of the rinsing and cleaning cycles and the composition of the coating baths, among other factors.



© Helmut Fischer GmbH Institute for Electronics and Measurement Technology Equipped with four measuring cells, each with separate inlets and outlets

Thanks to the standard equipment of the measuring device with four inline measuring cells and separate inlets and outlets, a separate measuring cell is available for each galvanic bath to be monitored. This considerably simplifies the bath feed, eliminates the need for valves and prevents contamination on both sides. In addition, the component materials for each channel can be specially adapted to the customer's electrolyte if required.

### For smooth workflows in your production process

The FISCHERSCOPE<sup>®</sup> XAN<sup>®</sup> LIQUID ANALYZER is connected to the electroplating bath either directly via a bath pump on the device side or via a ring line on the customer side. As soon as the solution to be analysed is fed to the flow-through measuring cell in the device, the metal content is checked fully automatically by means of X-ray fluorescence measurement. The measured values are displayed in real time on a screen (HMI) or exported directly to the higher-level control system via a fieldbus coupler. The special feature here is that the measurement data can be displayed simultaneously on other external monitors, for example directly on the electroplating bath. The operator of the system therefore

<sup>&</sup>lt;sup>1</sup> In a test setup under controlled laboratory conditions with a typical zinc-nickel solution, a lifetime of the measuring cell of over one year was achieved. The lifetime can vary and is individually dependent on the frequency of the rinsing and cleaning cycles, the composition of the coating baths and their temperatures, and the ambient conditions.

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always has the current measured values in view and can react quickly in the event of deviations, even if the measuring device is located in a separate room.



© Helmut Fischer GmbH Institute for Electronics and Metrology Large touchscreen with intuitive user interface, additional monitors directly at the respective electroplating bath

#### Market-leading measuring performance

In order to compare an XRF measurement with a classic wet chemical analysis (titration), measurements were carried out on a typical zinc-nickel bath using both methods. A typical zinc-nickel bath consists of 5 - 15 g/l zinc and 0.5 - 2 g/l nickel. From 10 measurements carried out with the FISCHERSCOPE<sup>®</sup> XAN<sup>®</sup> LIQUID ANALYZER with a measuring time of 10 s each, the following measurement results were obtained for the zinc concentration and the nickel concentration.



Fig. 1: Measurement results for zinc concentration, determined by titration and measured with the FISCHERSCOPE® XAN® LIQUID ANALYZER



Fig. 2: Measurement results for nickel concentration, determined by titration and measured with the FISCHERSCOPE® XAN® LIQUID ANALYZER

The measurement results for the determination of the zinc and nickel concentrations show close agreement with each other. Determining the metal concentration of an electroplating bath using XRF measurement is therefore very comparable to titration in terms of measurement precision.

In order to further demonstrate the high-performance measuring behaviour of the inline XRF measuring device, a further series of measurements was carried out on the zinc-nickel

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bath over a period of 48 hours using the FISCHERSCOPE<sup>®</sup> XAN<sup>®</sup> LIQUID ANALYZER. The measurement results are shown in Figure 3 and Figure 4.

Fig. 3: Measurement results for zinc concentration, measured with the FISCHERSCOPE® XAN® LIQUID ANALYZER over a period of 48 h



Fig. 4: Measurement results for nickel concentration, measured with the FISCHERSCOPE® XAN® LIQUID ANALYZER over a period of 48 h

The results demonstrate that the FISCHERSCOPE<sup>®</sup> XAN<sup>®</sup> LIQUID ANALYZER delivers very good repeatability. With a measurement time of 10 s per measurement, a mean value of 6.82 g/l was determined for the zinc concentration, with a coefficient of variation of only 0.26 %. A mean value of 1.26 g/l was determined for the nickel concentration with a

measuring time of 10 s per measurement and a coefficient of variation of 0.71 %. This means that the zinc-nickel bath is within the specified tolerances.

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

Table 1: Overview of the mean value, standard deviation and coefficient of variation of the zinc and nickel concentrations

The FISCHERSCOPE<sup>®</sup> XAN<sup>®</sup> LIQUID ANALYZER proves to be the optimum solution for efficient inline monitoring of metal concentrations in electroplating baths. The XRF measuring device not only scores points with its maximum measuring precision and repeatability of the measurement results. The fully automated and continuous analysis of up to four electroplating baths using four separate, long-life measuring cells eliminates the need for time-consuming and cost-intensive manual spot checks. The FISCHERSCOPE<sup>®</sup> XAN<sup>®</sup> LIQUID ANALYZER provides measurement data in real time and thus enables quality control in electroplating plants that is significantly faster and considerably more economical. This enables electroplating companies to ensure the consistently high quality of their electroplated coatings.



#### Have we piqued your interest?

Find out more about the FISCHERSCOPE® XAN® LIQUID ANALYZER!

Our Fischer experts will be happy to provide you with further information and personalised application consulting. **sales@helmut-fischer.com** 

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