



Application Note AN-T-223

Analysis of electroplating baths

Automatic pipetting with the OMNIS Sample Robot S

Electroplating processes are used in several different industry sectors to protect the surface quality of various products against corrosion or abrasion and significantly improve their working life. Depending on the bath composition, the outcome of this sophisticated process can vary for example in the layer thickness. It is therefore essential to check the bath composition on a regular basis to ensure that the process is operating correctly.

Typical examples of electroplating baths include alkaline degreasing baths or acidic or alkaline baths containing metals e.g. copper, nickel, or chromium, or components like chloride and cyanide. It is crucial that

the chosen analysis technique fulfills high safety standards for these kinds of analyses and produces reliable results.

The OMNIS Sample Robot system automatically pipettes and analyzes aggressive electroplating bath samples on different workstations. This reduces operator exposure to the often-harmful samples and increases sample throughput. The use of an OMNIS Sample Robot provides more reliable results in comparison to manual titration and is more time efficient, in particular due to the use of several workstations, where different parameters can be analyzed in parallel.

SAMPLE AND SAMPLE PREPARATION

In this application note, model substrates which are often found in common electroplating baths were prepared and then analyzed with the described setup:

0.5 mol/L CuSO_4 solution in 0.5 mol/L H_2SO_4 , 0.5 mol/L NiCl_2 solution, and 1.0 mol/L NaOH solution.

EXPERIMENTAL

The entire process is fully automated, including the sample transfer via pipette, the addition of water or auxiliary solutions, the rinsing of the sensor and

titration beaker, as well as removing the analyzed sample by the pumps. The only manual action is the filling of the beaker with the sample.

INSTRUMENTATION

The setup consists of two OMNIS Sample Robot S with four Pick&Place modules and two OMNIS pipetting equipments, allowing fast analysis of multiple parameters at the same time. The OMNIS Titrators, Dosing Modules, as well as the 846 Dosing Interface with 800 Dosing are equipped with various

titrants as well as auxiliary solutions, which are all dosed automatically.

Small volumes of the sample can be automatically transferred with the pipetting equipment, minimizing any human contact with the hazardous bath constituents.



Figure 1. OMNIS Sample Robot S with an OMNIS Titrator and three Dosing Modules. Not pictured: additional OMNIS Sample Robot with Titrator and Dosing Modules as well as required Dosing Interface and Dosinos.

Table 1. Summarized results of the mean value (n = 6) of the various electroplating bath samples.

Sample	Content in mol/L	Relative standard deviation
CuSO ₄ in H ₂ SO ₄	0.4790 H ₂ SO ₄ 0.5004 Cu(II)	0.05% 0.26%
NiCl ₂	0.9985 Cl ⁻ 0.5074 Ni(II)	0.22% 0.28%
NaOH	1.0004	0.17%

RESULTS

Low relative standard deviations for the different sample analyses show excellent reproducibility and demonstrate the outstanding accuracy of the pipetting equipment. A direct comparison between

various sample volumes show that even with 0.3 mL of pipetted sample, reliable and accurate results were obtained.

CONCLUSION

The OMNIS Sample Robot S equipped with the pipetting equipment is a fast, safe, and reliable setup to automate analysis of electroplating baths.

Moreover, multiple parameters of a single sample can be easily determined in one run.

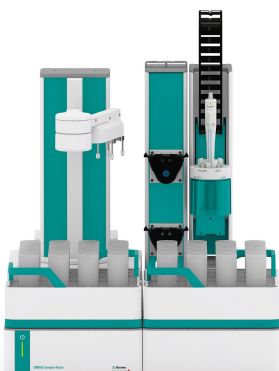
Internal reference: AW TI CH-1320-042021

CONTACT

Metrohm SA
1 Bridle Close
2052 Woodmead

sales@metrohm.co.za

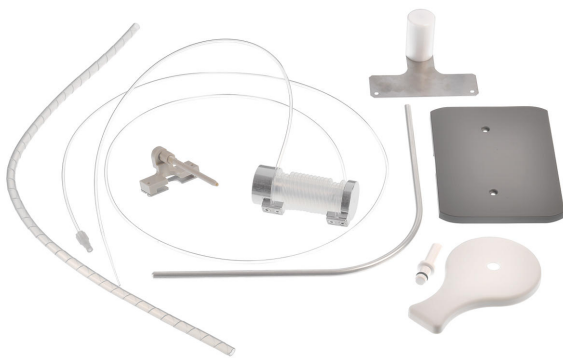
CONFIGURATION



OMNIS Sample Robot S Pick and Place

OMNIS Sample Robot S with a "Peristaltic" (2-channel) pump module and a Pick&Place module in addition to extensive accessories for the direct transition to fully automatic titration. The system provides space in two sample racks for 32 sample beakers of 120 mL each. This modular system is supplied completely installed and can thus be put into operation in a very short time.

The system can also be extended upon request to include two additional peristaltic pumps and another Pick&Place module, thus doubling the throughput. If additional workstations are required, then this Sample Robot is already able to be expanded to become an L-sized OMNIS Sample Robot, thus enabling samples from seven racks to be processed in parallel on up to four Pick&Place modules and quadrupling the sample throughput.



OMNIS pipetting equipment

Complete accessory set for converting the OMNIS Sample Robot Pick&Place into a version with pipetting options. The set can be mounted on all versions of the OMNIS Sample Robot (S,M and L).



OMNIS Basic Titrator without stirrer

Innovative, modular potentiometric OMNIS Titrator for endpoint titration. Thanks to 3S Liquid Adapter technology, handling chemicals is safer than ever before. The titrator can be freely configured with measuring modules and cylinder units and can have a stirrer added as needed. If required, the range of functions of the OMNIS Basic Titrator can be extended with a corresponding software function license.

- Control via PC or local network
- Can be supplemented with magnetic stirrer and/or rod stirrer
- Various cylinder sizes available: 5, 10, 20 or 50 mL
- Connection option for up to four additional titration or dosing modules for additional applications or auxiliary solutions
- Liquid Adapter with 3S technology: Safe handling of chemicals, automatic transfer of the original reagent data from the manufacturer

Measuring modes and software options:

- Endpoint titration: "Basic" function license
- Endpoint and equivalence point titration (monotonic/dynamic): "Advanced" function license
- Endpoint and equivalence point titration (monotonic/dynamic) with parallel titration: "Professional" function license



OMNIS Dosing Module without stirrer

Dosing module for connection to an OMNIS Titrator for extending the system to include an additional buret for titration/dosing. Can be supplemented with one magnetic stirrer or rod stirrer for use as separate titration stand. Freely selectable cylinder unit with 5, 10, 20 or 50 mL.



846 Dosing Interface

USB-capable control unit for connecting a maximum of four 800 Dosinos or 805 Dosimats for dosing and Liquid Handling tasks. A Touch Control or the connection to a PC with OMNIS Software, **tiamo**TM, MagIC Net, viva, or 797 VA Computrace is required for operation.



800 Dosino

The 800 Dosino is a drive with write/read hardware for intelligent Dosing Units. With fixed cable (length 150 cm).