Application Note AN-T-228

Determination of aluminum and zirconium in antiperspirants

Consecutive complexometric titrations with the Optrode to determine Zr and Al in one beaker

Antiperspirants contain zirconium aluminum chlorohydrates as active ingredients. Currently, the zirconium aluminum glycine (ZAG) complex is mainly used because it has been shown to be more effective. To evaluate the product quality, it is necessary to control the amount of both aluminum and zirconium in the ZAG complex. This determination can be done by complexometric titrations under different conditions according to the US Pharmacopeia (USP).

USP currently describes the assay of zirconium aluminum chlorohydrate complexes by two manual complexometric titrations after a laborious sample preparation procedure (digestion). This Application Note presents a complementary method that allows a consecutive determination after the sample preparation (digestion) of both metal ions in one beaker with an optical sensor and xylenol orange as an indicator.

SAMPLE AND SAMPLE PREPARATION

As samples, zirconium and aluminum standards were pipetted into a beaker and diluted with water. A drop of xylenol orange was added as color indicator.

EXPERIMENTAL

The pH of the sample was adjusted to pH 1 with 10 mL of buffer solution (pH 1). The sample was then titrated directly with EDTA (0.1 mol/L) on an OMNIS Titrator (Figure 1) to determine the Zr content (Figure 2). The equivalence point was reached once the solution turned from pink to yellow, detected by the Optrode at a wavelength of 574 nm. Afterward, 20 mL of acetate buffer (pH 4.7) and 15 mL of EDTA (0.1 mol/L) was added, followed by the back-titration of excess of EDTA with $Bi(NO_3)_3$ (0.05 mol/L). The equivalence point was reached once the solution turned from yellow to purple, detected by the Optrode at a wavelength of 574 nm. This corresponds to the Al content (Figure 3).

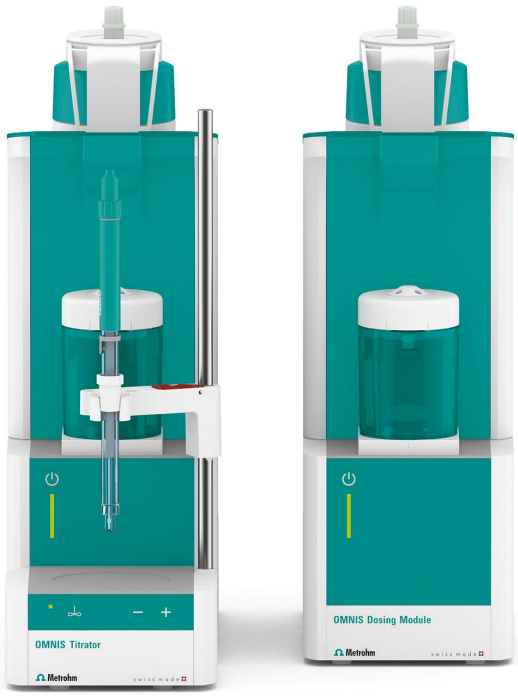

Figure 1. OMNIS Titrator with an OMNIS Dosing Module.

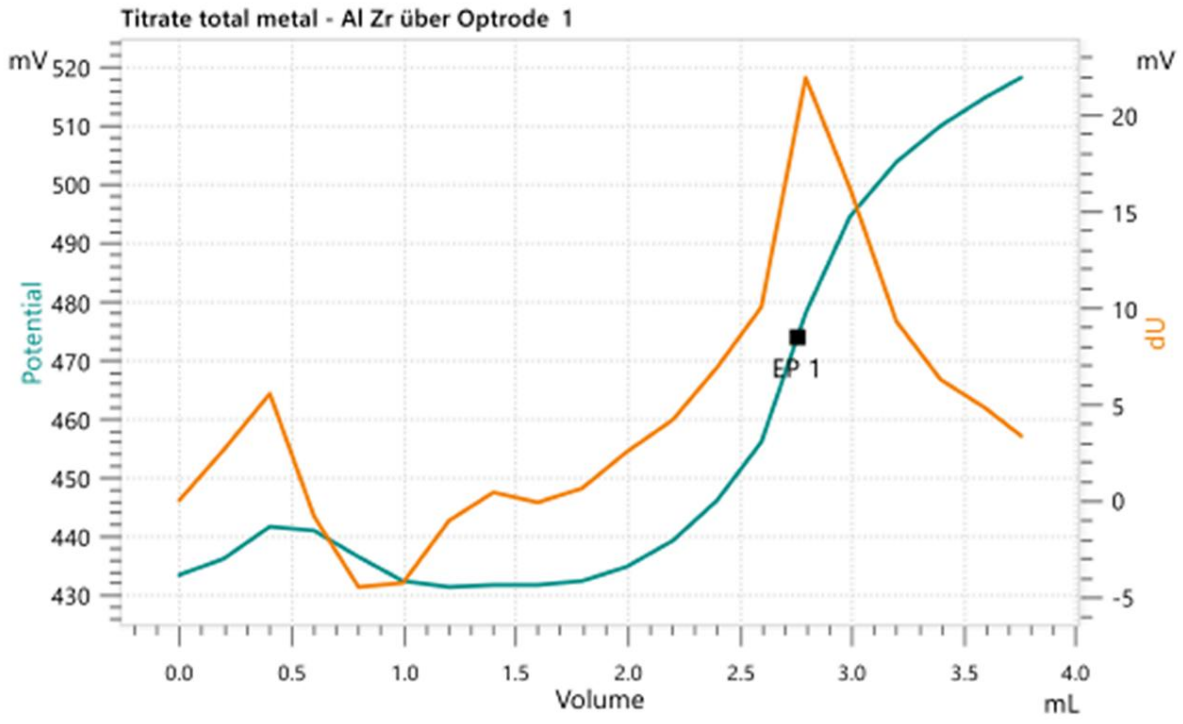

Figure 2. Example titration curve for Zr determination at pH 1 with EDTA as titrant.

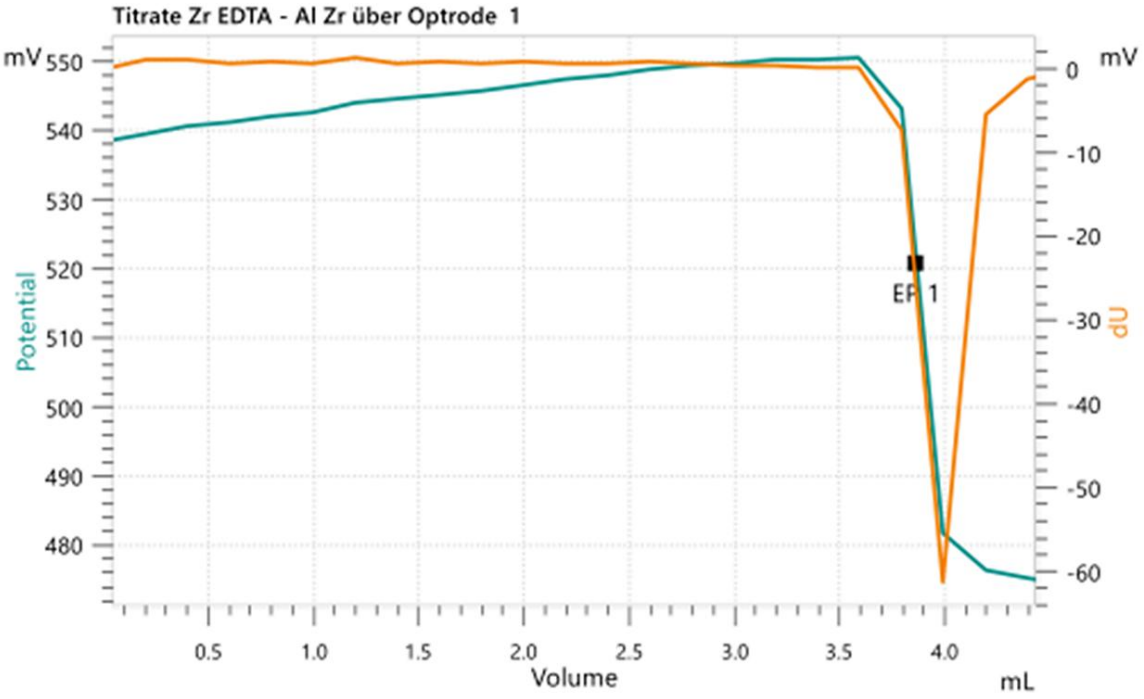

Figure 3. Example titration curve of the Al determination at pH 4.7 with bismuth nitrate as titrant.

Table 1. Summarized results for a mixture of Zr and Al standards (n = 3).

Metal	Mean (mg/mL)	RSD (%)	Recovery (%)
Zr	4.5015	0.69	98.9
Al	1.3289	2.13	97.3

RESULTS

The results presented in Table 1 were obtained for already liberated metal ions (i.e., Zr and Al standards) made from zirconyl chloride and aluminum chloride.

CONCLUSION

The analysis of both aluminum and zirconium ions can be realized successively on an OMNIS system. OMNIS allows fully automatic determinations, eliminating the need for extra laboratory work, saving time and effort.

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CONFIGURATION

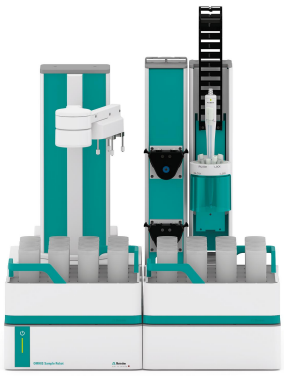
OMNIS Professional Titrator with magnetic stirrer

Innovative, modular potentiometric OMNIS Titrator for stand-alone operation or as the core of an OMNIS titration system for endpoint titration and equivalence point titration (monotonic/dynamic). Thanks to 3S Liquid Adapter technology, handling chemicals is more secure than ever before. The titrator can be freely configured with measuring modules and cylinder units and can have a rod stirrer added as needed. Including "Professional" function license for parallel titration with additional titration or dosing modules.

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

Measuring modes and software options:

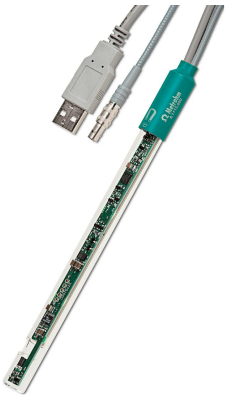
- Endpoint titration: "Basic" function license
- Endpoint and equivalence point titration (monotonic/dynamic): "Advanced" function license
- Endpoint and equivalence point titration (monotonic/dynamic) with parallel titration: "Professional" function license



OMNIS 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.



Optrode

Optical sensor for photometric titrations offering 8 different wavelengths. The wavelength can be switched using the software (tiamo 2.5 or higher) or with a magnet. The glass shaft is completely solvent-resistant and easy to clean. For example, this space-saving sensor is suitable for:

- Non-aqueous titrations in accordance with USP or EP
- Determinations of carboxyl end groups
- TAN/TBN in accordance with ASTM D974
- Sulfate determination
- Fe, Al, Ca in cement
- Water hardness
- Chondroitin sulfate in accordance with USP

The sensor is not suitable for determinations of concentrations via measurement of color intensity (colorimetry).