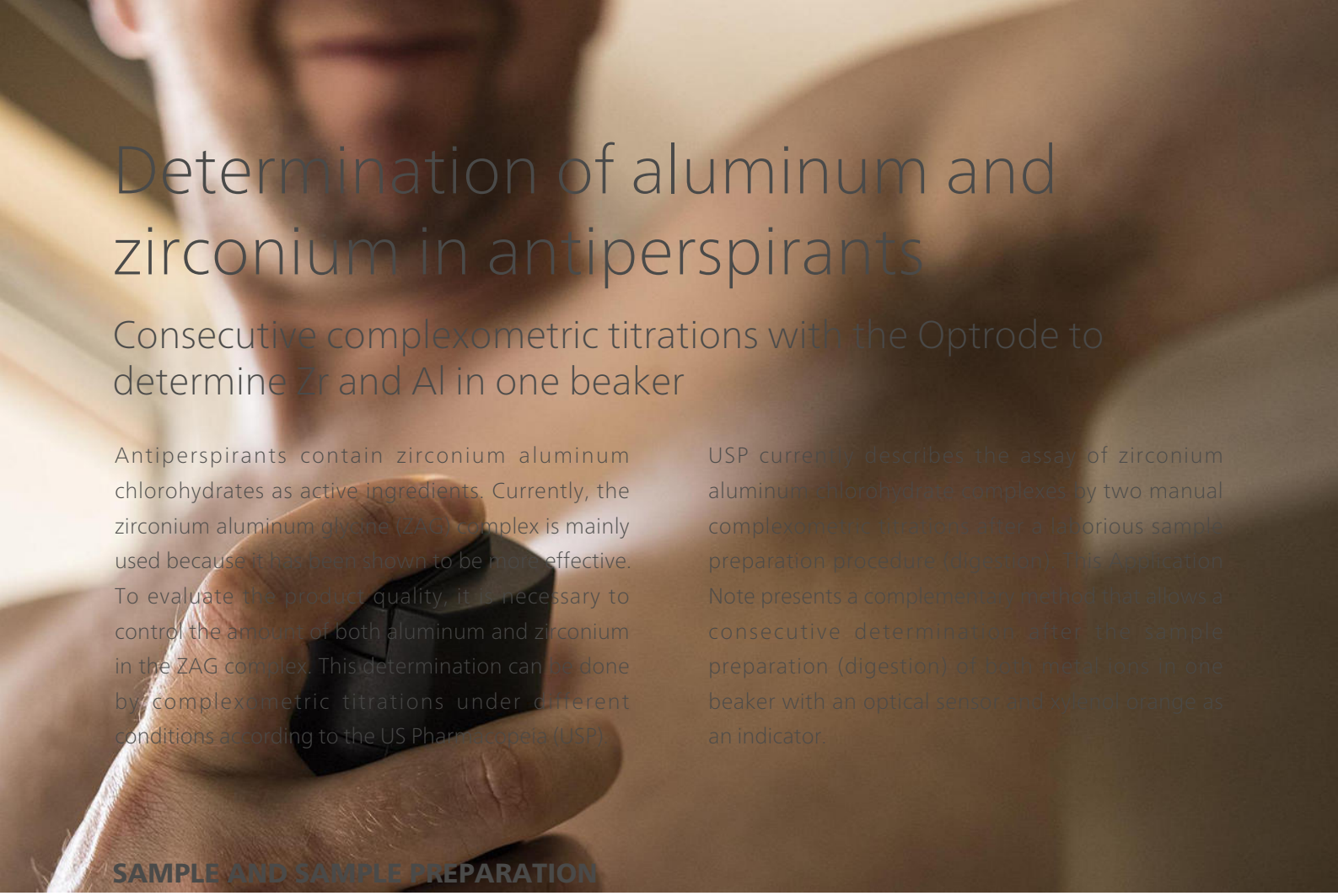

Determination of aluminum and zirconium in antiperspirants

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).

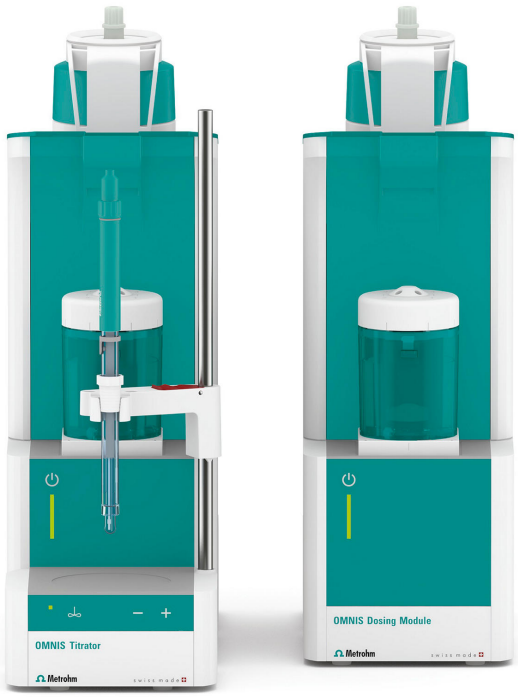

Figure 1. OMNIS Titrator with an OMNIS Dosing Module.

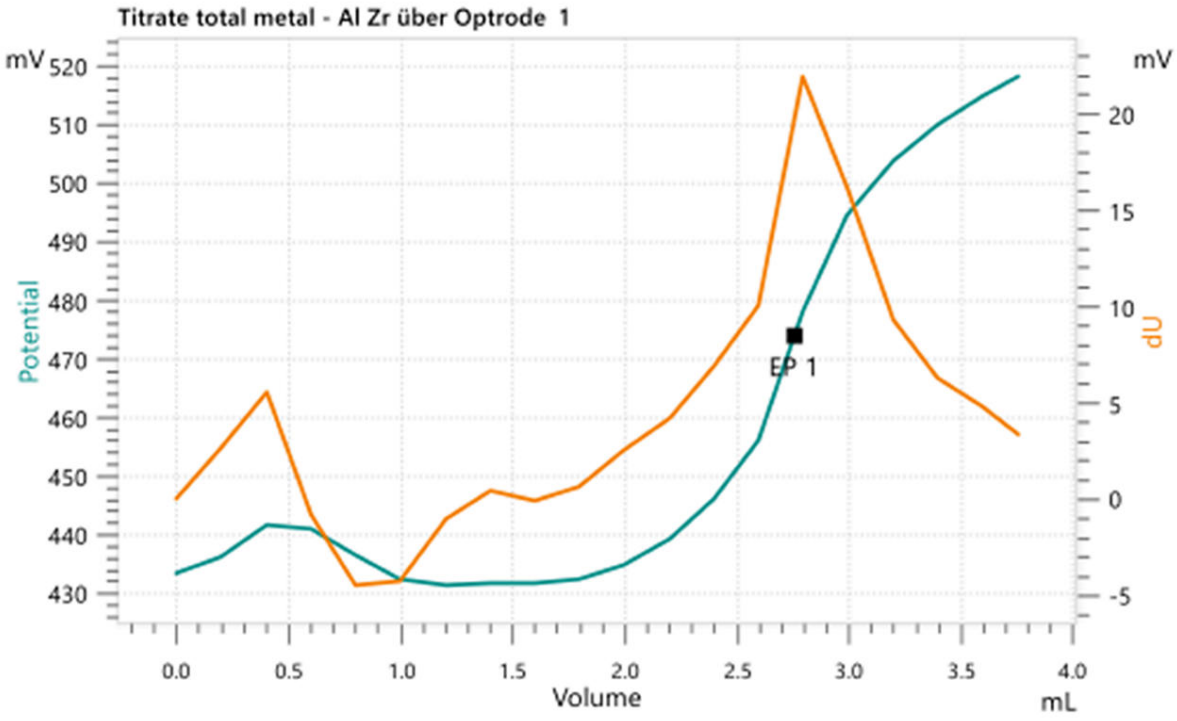

Figure 2. Example titration curve for Zr determination at pH 1 with EDTA as titrant.

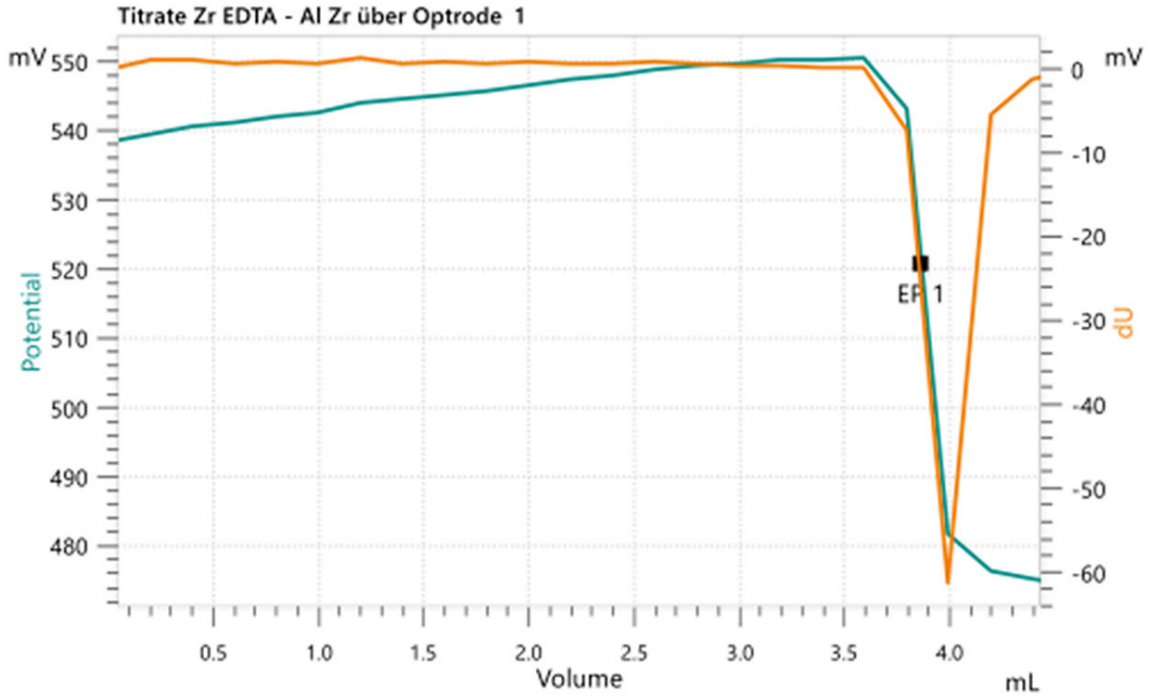

Figure 3. Example titration curve of the Al determination at pH 4.7 with bismuth nitrate as titrant.

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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