

止汗剂中铝和锆的测定

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).

SAMPLE AND SAMPLE PREPARATION

As samples, zirconium and aluminum standards were pipetted into a beaker and diluted with

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.

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 $\text{Bi}(\text{NO}_3)_3$ (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**).

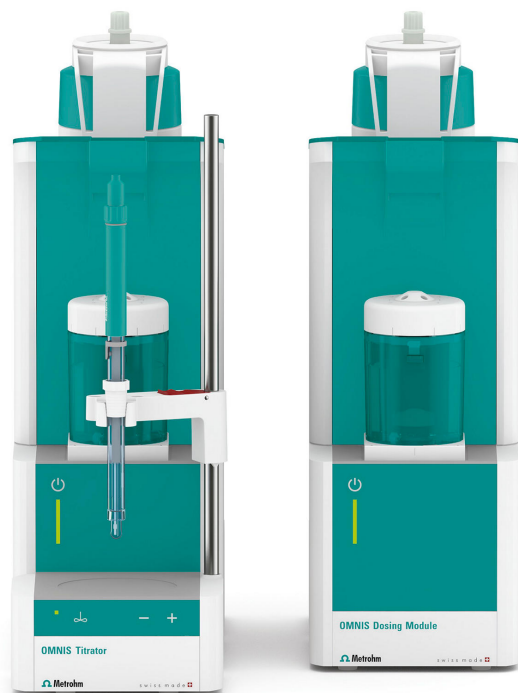


Figure 1. OMNIS Titrator with an OMNIS Dosing Module.

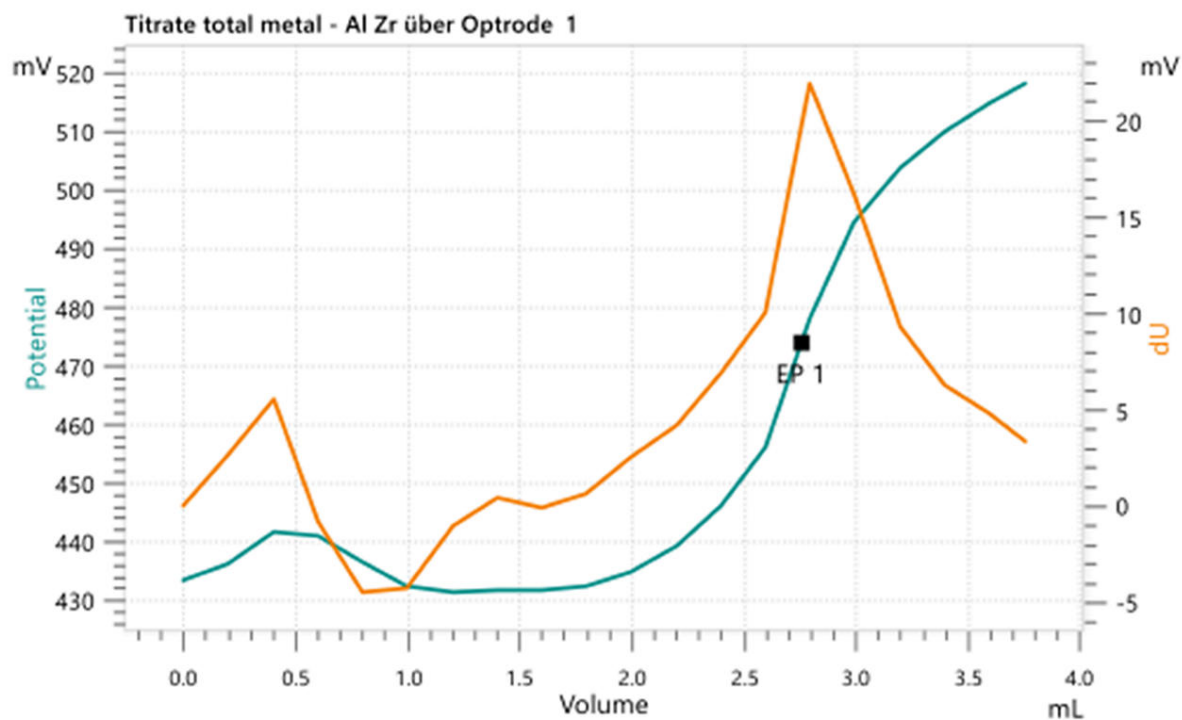


Figure 2. Example titration curve for Zr determination at pH 1 with EDTA as titrant.

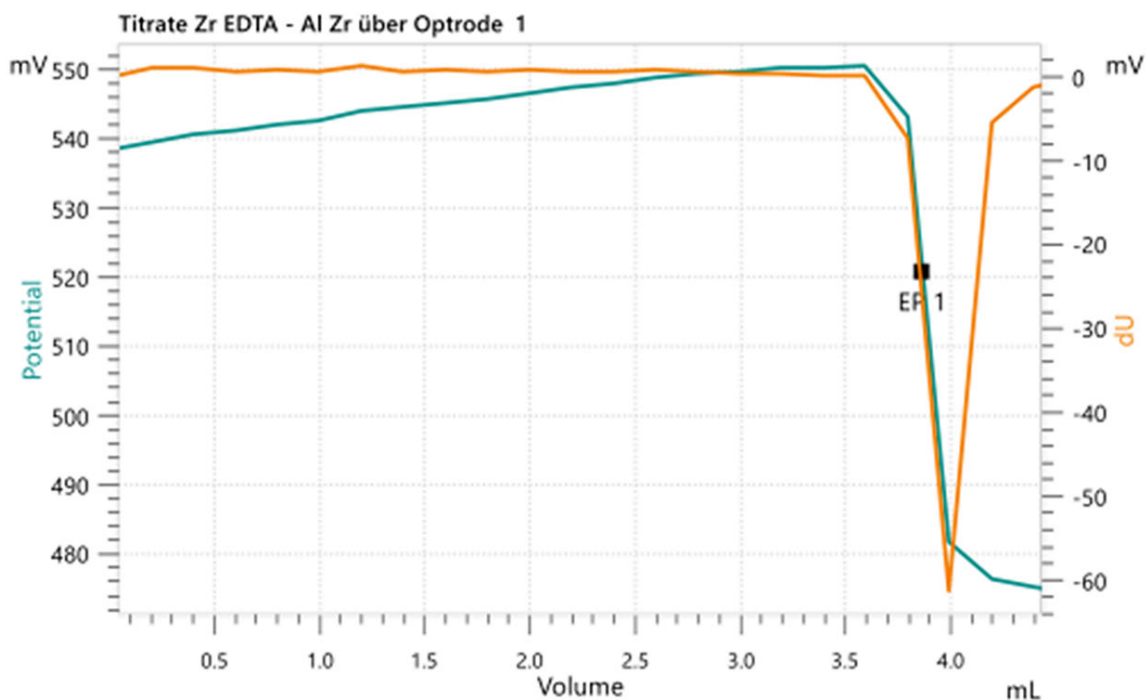


Figure 3. Example titration curve of the Al determination at pH 4.7 with bismuth nitrate as titrant.

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



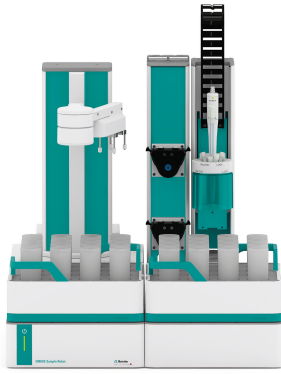
OMNIS Professional Titrator

创新型、模块式电位分析 OMNIS Titrator,适于独立运行或作为 OMNIS 滴定系统的核心元件运行,用于终点和等当点滴定(单一/动态)。由于采用 3S OMNIS Liquid Adapter 技术,处理化学品从未像现在一样安全。可以使用测量模块和计量管单元自由配置滴定仪,并在需要时扩展一台棒式搅拌器。包括用于使用其他滴定或加液模块平行滴定的“Professional”功能许可证。

- 通过计算机或本地网络控制
- 可以为其他应用或辅助溶液另外连接最多四个滴定模块或加液模块
- 棒式搅拌器的连接方式
- 可提供不同大小的计量管:5、10、20 或 50 mL
- 采用 3S 技术的 OMNIS Liquid Adapter:安全处理化学品,自动传输生产厂家的原始试剂数据

测量模式和软件选项:

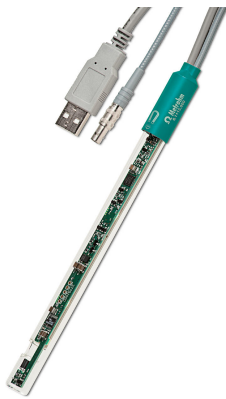
- 终点设定滴定:“Basic” 功能许可证
- 终点和等当点滴定(单一/动态):“Advanced” 功能许可证
- 终点和等当点滴定(单一/动态),包括 5 路平行滴定:“Professional” 功能许可证



OMNIS Sample Robot S Pick and Place

OMNIS Sample Robot S 具有一个“蠕动”泵模块(2通道)和一个 Pick&Place 模块以及大量附件,可直接进入全自动滴定。此系统具有两个样品盘位置,可用于 32 个 120 mL 的样品盘。此模块化系统供货时已完全安装完毕,因此可在极短时间内投入运行。

该系统也可根据需要扩展配备另外两台蠕动泵以及多加一个 Pick&Place 模块,由此使通量翻倍。如果需要更多工作台,则可将此 Sample Robot 扩展为 L 规格款型的 OMNIS Sample Robot,由此可使七个样品盘的样品在多达四个 Pick&Place 模块上并行处理,将样品通量扩大四倍。



Optrode

有 8 种可用波长的光度滴定用光学传感器。可以通过软件控制(tiamo 2.5 及以上版本)或通过磁铁来进行波长切换。该玻璃鞘对溶剂耐受,并且易于清洁。这种节省空间的传感器适用于,例如:

- 按照 USP 或 EP 的非水溶性滴定
- 羧基端基的测定
- TAN/TBN 根据 ASTM D974
- 硫酸测定
- 混凝土中的 Fe、Al、Ca
- 水硬度
- 根据 USP 的硫酸软骨素

传感器不适合通过测量颜色强度(比色法)来测定浓度。