Application Note AN-T-249

Analysis of rare earth metals based on ISO 23597

Fast and accurate photometric determination of rare earth elements (REEs) with the Optrode

Once extracted and separated from deposits, rare earth elements (REEs) are refined and sold as metal ingots, metal powders, rare earth oxides (REOs), alloys, or salts. REOs usually have a purity level of over 97% while ingots and powders are at least 99%.

The mass fraction of rare earth metals in these products is an important quality parameter and a key price and usability determinant. The pure REEs are typically dissolved in acid, after which their purity is analyzed using various methods.

One of the most important of these methods is titration using xylenol orange as an indicator (ISO 23597).

Titration is an absolute method that offers many advantages over other established techniques such as atomic absorption spectroscopy (AAS). Titrations using the Optrode M2 have been shown to yield precise and reproducible results, with recovery rates of around 100% for rare earth elements.

INTRODUCTION

Photometric titration is a highly accurate, fast, and cost-effective method of determining the purity of rare earth metals. Other analysis methods like AAS require calibrations to be performed in advance. The standards used for this purpose are often analyzed by rare earth metals titration. Therefore, it makes sense to determine rare earth elements directly by titration rather than using a complex AAS method that also requires calibration.

The fully dissolved rare earth elements are mixed with xylenol orange (XO). This indicator forms a colored complex with the rare earth cation (Ln3+), which is then titrated with ethylenediaminetetraacetic acid (EDTA). Once all the metal ions have been complexed or titrated away, the color of this complex changes.

The following reactions offer a highly simplified description of the titration with a lanthanide cation:

$Ln^{3+} + XO \rightarrow Ln(XO)$

$Ln(XO) + EDTA \rightarrow Ln(EDTA) + XO$

Ln(XO) is red and XO is yellow. The Optrode can be used to determine this color change very precisely at a wavelength of 574 nm.

SAMPLE AND SAMPLE PREPARATION

Dissolved rare earth element standards in nitric acid were used as samples. These samples are very similar to the acid-dissolved REEs used in industry, e.g., those used for AAS determination. The method was performed based on ISO 23597 [1] as well as on the paper from S. J. Lyle et al [2]. This application is demonstrated on yttrium, samarium, and dysprosium standard solutions. No sample preparation is required.

EXPERIMENTAL

The determinations are carried out on an OMNIS Professional Titrator equipped with OMNIS Dosing Modules (**Figure 1**) and an Optrode M2.

An appropriate amount of sample is weighed into the titration beaker, and acetate buffer as well as xylenol orange indicator solution are added. The solution is titrated until after the first equivalence point with standardized EDTA solution.

Figure 1. When equipped with an Optrode M2, the OMNIS Professional Titrator with OMNIS Dosing Modules (shown here) is an ideal setup for rare earth metal determination.

RESULTS

As shown in Table 1, the results present high recovery rates and confirm the robustness of photometric titration. An exemplary titration curve of yttrium standard solution is given in Figure 2.

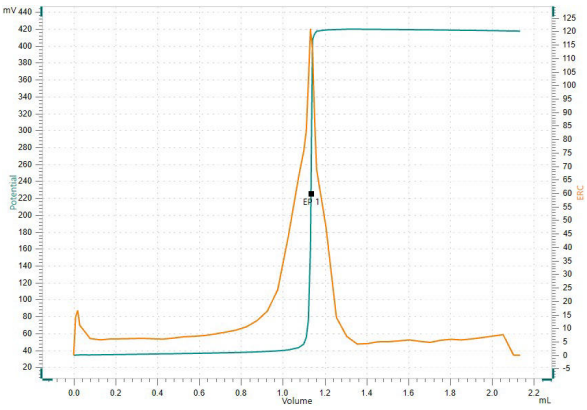

Figure 2. Photometric titration of yttrium standard solution using the Optrode M2.

Table 1. Results of the photometric titration of rare earth elements with the Optrode M2 (n = 6).

Sample (n = 6)	Content (g/L)	Recovery (%)
Yttrium	9.98	100.0
Samarium	10.10	100.6
Dysprosium	10.08	100.9

CONCLUSION

In this Application Note, the purity of rare earth elements was photometrically determined with the Optrode M2. This method provides a compelling, precise and cost-effective alternative to standard analytical techniques, like AAS, for determining the purity of rare earth elements. As it is an absolute method, complex calibration is not required, making titration particularly attractive to customers.

Thanks to the individually adjustable wavelength of the Optrode and the reliable OMNIS Software, the REE determination based on ISO 23597 can be achieved for all known rare earth elements.

REFERENCES

1. Rare Earth — Determination of Rare Earth Content in Individual Rare Earth Metals and Their Oxides — Titration Method; ISO 23597:2023; 2023; p 10.
2. Lyle, S. J.; Rahman, Md. M. Complexometric Titration of Yttrium and the Lanthanons—I: A Comparison of Direct Methods. Talanta 1963, 10 (11), 1177–1182. DOI:10.1016/0039-9140(63)80170-8

CONTACT

瑞士万通中国
北京市海淀区上地东路1号
院1号楼7层702
100085 北京
marketing@metrohm.com.cn

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 Dosing Module

用于与 OMNIS Titrator 滴定仪相连的加液模块,以扩展额外用于滴定/加液的滴定管。可以扩展磁力搅拌器和/或螺旋搅拌器,以作为单独的滴定台使用。可自由选择 5、10、20 或 50 mL 计量管单元。

Optrode M2

有 8 种可用波长的光度滴定法用光学传感器。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。该玻璃鞘对溶剂完全耐受,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。