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.

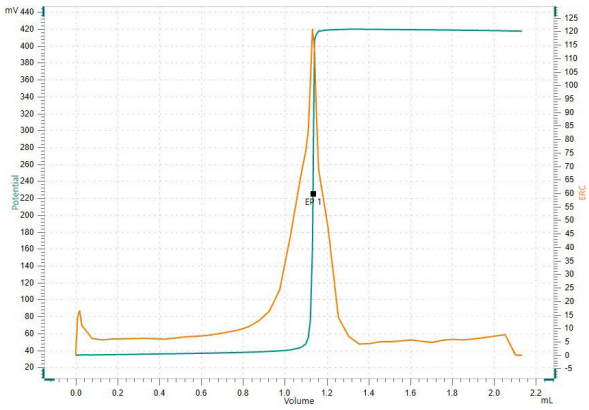

Figure 2. Photometric titration of yttrium standard solution using the Optrode M2.

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

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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.

- Actuation 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: 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 5-way 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.

Optrode M2

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).