



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 (Ln^{3+}), 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:



$\text{Ln}(\text{XO})$ 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 con agitador magnético

El OMNIS Titrator es un aparato potenciométrico, modular e innovador para el funcionamiento en modo stand-alone o como elemento central de un sistema de titulación OMNIS para la titulación a punto final y a punto de equivalencia (monótona/dinámica). Gracias a Liquid Adapter con tecnología 3S, este titulador resulta más seguro que nunca para el manejo de productos químicos. El titulador se puede configurar libremente con módulos de medida y unidades de cilindro y, si es necesario, se le puede añadir un agitador de varilla. Incluye la licencia funcional "Professional" para la titulación en paralelo con módulos de titulación o dosificación adicionales.

- Control a través de ordenador o red local
- Posibilidad de conexión de hasta 4 módulos de titulación o dosificación más para otras aplicaciones o soluciones auxiliares
- Posibilidad de conexión de un agitador de varilla
- Diferentes tamaños de cilindro disponibles: 5, 10, 20 o 50 mL
- OMNIS Liquid Adapter con tecnología 3S: manejo seguro de productos químicos, transferencia automática de los datos originales del reactivo del fabricante

Modos de medida y opciones de software:

- Titulación a punto final: licencia funcional "Basic"
- Titulación a punto final y titulación a punto de equivalencia (monótona/dinámica): licencia funcional "Advanced"
- Titulación a punto final y titulación a punto de equivalencia (monótona/dinámica) con titulación quintuple en paralelo: licencia funcional "Professional"



OMNIS Dosing Module sin agitador

Módulo de dosificación para la conexión de un titulador OMNIS para incluir una bureta adicional de titulación/dosificación. Se puede ampliar con un agitador magnético o de varilla para su uso como stand de titulación independiente. Libre selección de la unidad de cilindro con 5, 10, 20 o 50 mL.



Optrode M2

Sensor óptico para titulaciones fotométricas con 8 longitudes de onda disponibles. La longitud de onda se puede cambiar por software (a partir de tiamo 2.5) o con un imán. El mango de vidrio es totalmente resistente a los disolventes y fácil de limpiar. El sensor permite ahorrar espacio y es adecuado, por ejemplo, para:

- Titulaciones no acuosas según USP o EP
- Determinaciones de grupos carboxilos finales
- TAN/TBN según ASTM D974
- Determinación de sulfato
- Fe, Al, Ca en cemento
- Dureza del agua
- Sulfato de condroitina según USP

El sensor no es adecuado para determinar las concentraciones midiendo la intensidad del color (colorimetría).