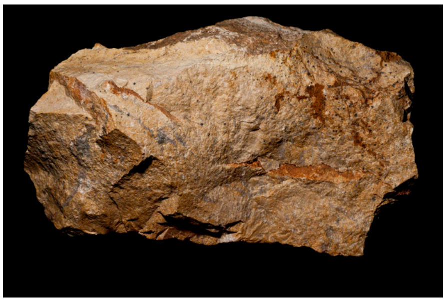
Application note | 41526

# Analysis of rare earth elements in clay using XRF and XRD



**Bastnäsite is a mineral that usually contains La, Ce and Y.**

## Introduction

Rare earth elements (REEs) are 17 elements used in dozens of high-tech applications. The global demand for REEs has surged in recent years, driven by advancements in technology and the transition towards green energy solutions. REE magnets are compact, efficient, and hard to replace. Electric vehicles (EVs) and wind power are the main growth industries for these use cases.

Although rare earth elements are relatively abundant in the earth's crust, they are rarely concentrated into mineable ore deposits. REEs are associated with alkaline rocks, carbonatite (bastnäsite, monazite, parisite, and synchysite), pegmatite, and placer deposits (residual deposits formed from deep weathering) and are often discovered via geochemical exploration. They may also be absorbed in clay minerals or ion adsorption clays (IACs) such as kaolinite, illite and smectite. The ores of rare earth elements are mineralogically and chemically complex and commonly radioactive. REE mining is challenging because REEs are concentrated in more than one mineral, and each mineral requires a different costly extraction technology and mineral processing.

For companies operating in REE exploration and mining, the accuracy of geochemical analysis is critical to assess the viability of deposits, respectively targeting REE rich areas for extraction. The Thermo Scientific™ ARL™ X'TRA Companion X-ray Diffractometer detects and quantifies mineral phases that contain REEs such as kaolinite, smectite, monazite and bastnasite. Thermo Scientific™ ARL™ PERFORM'X WDXRF Spectrometer on the other hand provides fast, accurate major/minor oxides analysis on the REE ores.

**Figure 1. ARL X'TRA Companion X-ray diffraction system.**

**Figure 2. ARL PERFORM'X wavelength dispersive XRF spectrometer.**

## Instrument and software

The ARL X'TRA Companion X-Ray Diffractometer (Figure 1) is a simple, easy-to-use benchtop instrument for routine phase analysis as well as more advanced applications. The ARL X'TRA Companion XRD uses a θ/θ goniometer (160 mm radius) in Bragg-Brentano geometry coupled with a 600 W X-ray source (Cu or Co). The radial and axial collimation of the beam is controlled by divergence and Soller slits, while air scattering is reduced by a variable beam knife. An integrated water chiller is available as an option. Thanks to the state-of-the art solid state pixel detector (55x55 µm pitch), the ARL X'TRA Companion XRD provides very fast data collection and comes with single-click Rietveld quantification capabilities and automated result transmission to a LIMS (Laboratory Information Management System). Both capabilities are seamlessly integrated into Thermo Scientific™ SolstiX™ Pronto Instrument Control Software.

The ARL PERFORM'X series spectrometer (Figure 2) used in this analysis was a 2500-watt system. This system is configured with 6 primary beam filters, 4 collimators, up to nine crystals, two detectors and our 5GN+ Rh X-ray tube for best performance from ultra- light to heaviest elements thanks to its 50 microns Be window. This innovative, ultra-light X-ray tube fitted with a low current filament ensures an unequalled analytical stability month after month. When paired with industry-leading Thermo Scientific™ UniQuant™ Software the ARL PERFORM'X spectrometer provides the best limits of detection and accuracy in standardless analysis up to element Am.

## Experimental

For this experiment, NCS DC 86311 certified reference material (CRM) was used. This is a CRM for rare earth ore by NCS (China) with a certificate of elemental oxides.

For XRD study, the CRM was manually pressed in top loading sample cup and measured in reflection mode using Cu Kα (1.541874 Å) radiation for 20 minutes with sample spinning. (Figure 2). Rietveld refinements were carried out using Profex software.

For the WDXRF study, the CRM was prepared as pressed pellets for trace level analysis. Pressed pellets offer higher density and analytical precision for applications like geochemical analysis by XRF, as compared to loose powders. UniQuant standardless analysis was done with 20 seconds counting time on all analytical lines

## Results and discussion

### XRD results from ARL X'TRA Companion X-ray Diffratctometer

Qualitative (Figure 3) and quantitative (Table 1) XRD analysis of the REE CRM indicates a granite-weathering analogue relevant to ion-adsorption clays.

**Table 1. Phase quantities from Rietveld refinement of NCS DC 86311 CRM**

| Phase | Quantity (in wt%) |
|---|---|
| Quartz | 50.4 |
| Microcline | 30.8 |
| Smectite | 9.7 |
| Kaolinite | 3.8 |
| Orthoclase | 2.0 |
| Albite | 1.6 |
| Illite | 1.1 |
| Hematite | 0.5 |
| Goethite | 0.1 |

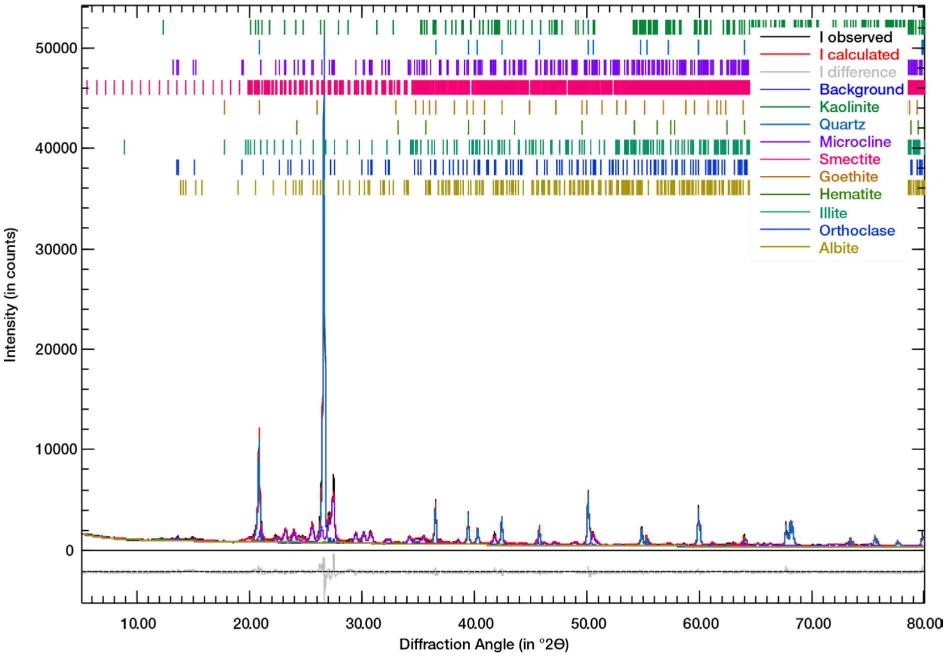



**Figure 3. Diffractogram of NCS DC 86311 CRM obtained using ARL X'TRA Companion XRD.**

## WDXRF results from ARL PERFORM'X spectrometer

Table 2 shows the analysis results of NCS DC 86311 CRM using UniQuant software compared to the reference results.

### Table 2. UniQuant analysis results vs reference results

| Compound | UniQuant (%) | Est. error on UniQuant software estimation | Reference results (certified %) |
|---|---|---|---|
| $SiO_2$ | 74.5 | 0.46 | 74.34 |
| $Al_2O_3$ | 14.71 | 0.11 | 14.65 |
| $K_2O$ | 4.96 | 0.15 | 4.92 |
| $Fe_2O_3$ | 1.14 | 0.08 | 1.13 |
| $Y_2O_3$ | 0.309 | 0.015 | 0.303 |
| $Na_2O$ | 0.131 | 0.012 | 0.155 |
| MgO | 0.0892 | 0.004 | 0.08 |
| $Rb_2O$ | 0.0662 | 0.0033 | 0.067 |
| $Dy_2O_3$ | 0.0367 | 0.0018 | 0.036 |
| $Gd_2O_3$ | 0.0259 | 0.0013 | (0.027) |
| $Yb_2O_3$ | 0.0232 | 0.0012 | 0.022 |
| $Er_2O_3$ | 0.0216 | 0.0011 | 0.022 |
| $Nd_2O_3$ | 0.0214 | 0.0011 | 0.022 |
| $TiO_2$ | 0.0159 | 0.0006 | (0.023) |
| $Cs_2O$ | 0.0154 | 0.0054 | 0.00178 |
| $Sm_2O_3$ | 0.0137 | 0.0007 | 0.015 |
| MnO | 0.0134 | 0.0007 | 0.016 |
| CaO | 0.01 | 0.0004 | (0.031) |
| $La_2O_3$ | 0.01 | 0.0005 | 0.011 |
| $P_2O5$ | 0.0069 | 0.0054 | (0.0025) |
| $Tb_4O_7$ | 0.0057 | 0.0007 | 0.0058 |
| $Pr_6O_{11}$ | 0.0042 | 0.0008 | (0.0045) |
| $ThO_2$ | 0.0036 | 0.0011 | 0.0039 |
| $CeO_2$ | 0.0031 | 0.0009 | 0.0035 |
| $Tm_2O_3$ | 0.0026 | 0.0006 | 0.0032 |
| $Sc_2O_3$ | 0.00057 | 0.00012 | (0.00089) |

( ) = for reference only

## Your benefits

The ARL X'TRA Companion XRD workflow delivers answers in a few minutes, quantifying the mineral phases available in the ion adsorption clay samples. Users are able to identify the type of mineral phases associated with REEs such as kaolinite, halloysite, smectite, illite. They are also able to verify the ion adsorption clay (IAC) mechanism due to absence of monazite/xenotime peaks & availability of kaolinite & smectite which supports ion-adsorbed REE.

ARL PERFORM'X WDXRF spectrometer together with UniQuant standardless analysis software enables the users to screen through REE ores for up to 79 elements without the need for conventional calibrations. Well suited for mining ore analysis which will have varying sample matrices. UniQuant software is fully calibrated and installed from the factory. Hence the ARL PERFORM'X spectrometer is ready to perform meaningful analysis of unknown samples directly after installation at the customer's site.

**Monazite is a phosphate mineral that contains rare earths elements.**

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