Appendix OM3: Determining peak and background position for the REE (with a focus on interferences)
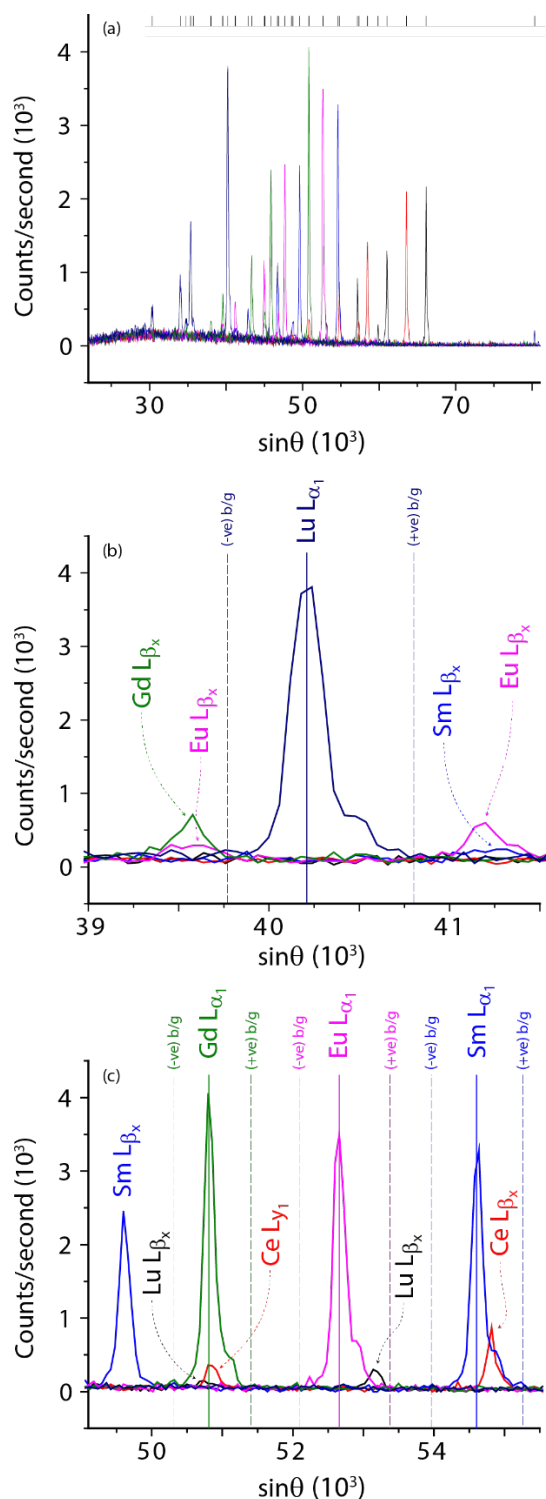
In order to determine peak positions and potential interferences on the REE present within experiments (i.e., La, Ce, Sm, Eu, Gd and Lu) full wavelength scans were carried out on the relevant $(REE^{3+})PO_4$ standards on the LLIF spectrometer. Scans involved a focused (1 μm) beam with an accelerating voltage of 15 kV and beam current of 40 nA (i.e., equivalent to analytical conditions for REE measurements made from ApREE experiments). The full wavelength scan was carried out from 22008 to 81948 sinθ, using a 60 sinθ step and 0.1 second dwell time over 1000 channels.

Of the elements measured in the study, suitable peak and background positions can be found for the $L\alpha_1$ peaks based on comparison of $(REE^{3+})PO_4$ wavelength scans. The one possible exception is the Gd $L\alpha_1$ peak at 50533 sinθ, which shows direct overlap with the Ce $L\gamma_1$. Using the starting composition of REE within experiments it is possible to model the overlap correction based on the relative concentrations using Virtual WDS by Reed and Buckley (1998; see Table Oms3t1). Where our background positions are generally higher than Reed and Buckley's, the peak positions used in this study are defined by scans made individually on the in-house instrument (see Fig. OMs3). This being the case, as the peak overlap correction in Virtual WDS (Reed and Buckley, 1998) is a measure of counts derived from other spectral components at the sinθ of analysis (i.e., this is not the same as a slope correction that pertains to background positions) it is possible to make an overlap correction. Calculated Virtual WDS corrections based on the experimental starting composition, shows a recommended overlap correction that is several orders of magnitude below values reported in Table OM3 and Tables OM1a-1b. In other words, the REE overlap corrections for experiments from this study are negligible.

Table OM3

| Element | Line | This study | | Reed and Buckley (1998) | | Recommended overlap correction |
		-ve b/ground	+ve b/ground	-ve b/ground	+ve b/ground	
La	$L\alpha_1$	600	800	300	400	0
Ce	$L\alpha_1$	660	660	300	400	+0.00008%
Sm	$L\alpha_1$	650	650	300	450	-0.00003%
Eu	$L\alpha_1$	560	720	300	450	+0.00004%
Gd	$L\alpha_1$	500	600	300	450	0
Lu	$L\alpha_1$	415	595	300	450	0

Note: overlap calculations assume a REE concentration based on ratios defined by experimental starting material, where La:Ce:Sm:Eu:Gd:Lu are 0.1:0.2:0.1:0.8:0.1:0.1 (by weight).

Figure OM3: WDS wavelength scans from $LaPO_4$, $CePO_4$, $SmPO_4$, $EuPO_4$, $GdPO_4$ and $LuPO_4$ standards measured on the LLIF spectrometer in this work. (a) Full LLIF wavelength scan of all standards, black lines at top of Figure correspond to absorption lines observed across all scans. (b) Focused wavelength scan over the Lu $L\alpha_1$ peak from 39000-41500 $sin\theta$, where solid line corresponds to the peak position and dashed curves correspond to background positions (other absorption lines are labeled for clarity). (c) Focused wavelength scans over the Gd, Eu and Sm $L\alpha_1$ peaks, background positions and peak labels as in previous Figures.