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Challenges in Electron Probe Micronalysis 60 Years after Castaing: Examples from Complex Uranium and Rare Earth Element Minerals from Northern Australian Ore Deposits

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Electron probe microanalysis (EPMA) has widely been considered a mature technique for decades, but the changing and complex chemical composition of many natural phases still requires extensive method development on a routine basis. It is frequently limited by issues like spectral interferences, matrix correction uncertainties, secondary fluorescence across grain boundaries, phase stability under the beam, and the availability of suitable standards for calibration and method validation.

Examples from Northern Australian ore deposits will be presented, such as the minerals uraninite UO_2 and florencite $REEAl_3(PO_4)_2(HO)_6$ occurring in the polymetallic deposit Coronation Hill in Northern Australia. It has an estimated resource of 3.5 Mt of more than 5 g/t Au, 0.2 g/t Pt, and 0.5 g/t Pd and contains numerous Ag, As, and Se minerals [1]. In addition to that, an *in situ* U resource at Coronation Hill is estimated at 350 kt of around 0.5% U. It may be part of the same mineralising system as deposits in the East Alligator River area, which currently supply about a quarter of the world's uranium. It has so far not been mined nor has it been fully tested or dated [2].

Both uraninite and florencite can contain a wide range of elements including rare earth elements (REE), Sr, Y, Th, Pb, Bi, Ca, As, and Fe at significant concentrations, depending on the geological context [3,4]. Also, REE patterns of uraninite can vary significantly and exhibit unusual shapes [3]. The main challenge for EPMA of these phases lies in resolving spectral interferences and positioning background measurements, especially due to the notorious cross-interferences between the REE as shown for Sm in Fig. 1. The impact of alternative background measurement techniques like mean atomic number background correction [5] and multi-position background measurement will be discussed.

Uraninite from Coronation Hill is enriched in Y (up to 1 wt%), Ca (up to 4 wt%), and heavy REE (up to 0.4 wt% Dy). It contains up to 11 wt% Pb, but isotopic Pb data from LA-ICP-MS indicates substantial Pb loss after crystallisation. Florencite is rich in LREE (up to 12 wt% Ce), Sr (up to 3 wt%), S, Ca, and Fe (all 0.8–1.5 wt%) and occurs in very fine-grained clusters sometimes exceeding 100 um in size as shown in Fig. 2. The clusters also contain quartz, apatite and Fe oxides. This, combined with the strong susceptibility of the phase to electron beam damage, strongly limits the analytical sensitivity in EPMA. The small grain size also requires careful consideration of interaction volume and secondary fluorescence. The results will be compared with LA-ICP-MS data and previously published data from other deposits in the region such as [4].

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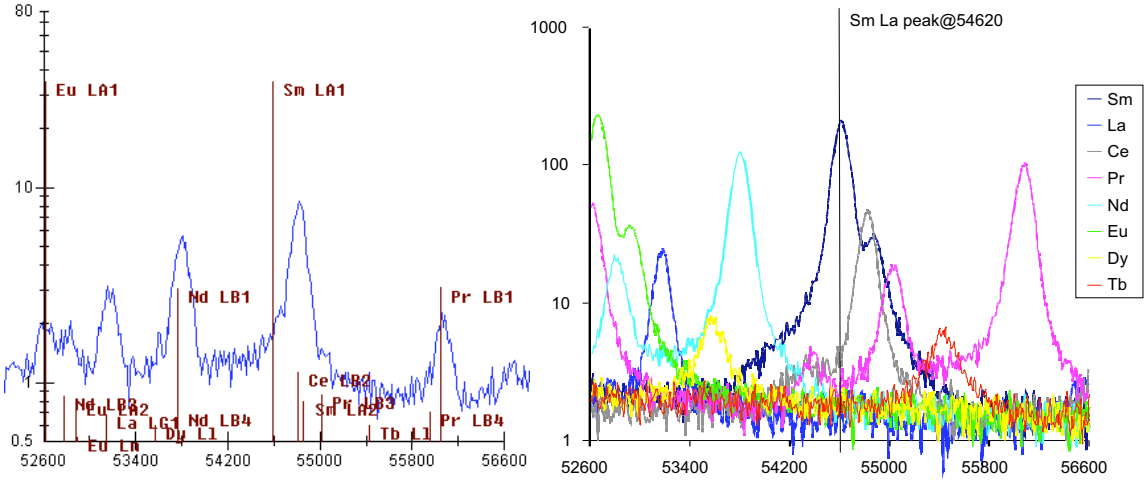

FIG. 1. LiF crystal wavelength scans in the region of Sm Lα on florencite from Coronation Hill (left) and various REE orthophosphates (right, [6]) to illustrate peak overlaps. x axis: spectrometer units (sinθ_10^5), y axis: x-ray intensity (cps).

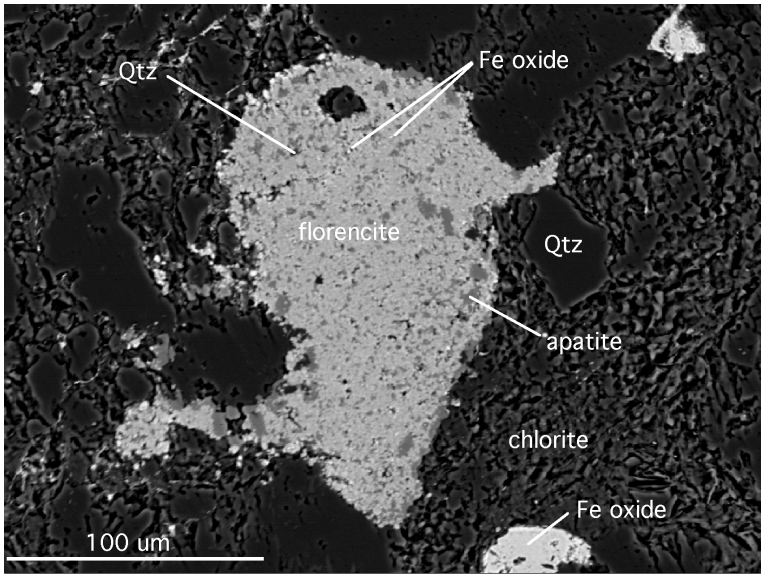

FIG. 2. Backscattered electron image of typical florencite cluster in sample 49-308B from Coronation Hill.

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