

# Quantitative Microchemical Analysis Using Electron Probe Microanalysis: A Study Featuring the Analysis of Irradiated FUTURIX-FTA U-Pu-Zr with Added Minor Actinides

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Electron Probe Microanalysis (EPMA) is a quantitative microchemical analysis technique that allows measurement of major and trace elements in a solid sample with a spatial resolution on the order of  $1\ \mu\text{m}^3$ . Analytical error for major elements is on the order of 1-2%, while trace element error is on the order of 10-20%. This instrument can be shielded for up to 3 Ci of  $^{137}\text{Cs}$ , thus allowing precise analysis of high irradiated nuclear fuel. Such an instrument was used for the microchemical analysis and characterization of FUTURIX-FTA fuel specimen "DOE1". This fuel was composed of 34.1U-28.3Pu-3.8Am-2.1Np-31.7Zr (where the preceding numbers indicate concentrations in weight %). The fuel was irradiated in the Phénix sodium fast reactor in France to a measured burnup of 9.5% fissions per initial heavy metal atom, and experienced a peak cladding temperature of 550°C. Results show that Zr concentration in the fuel center increased from its as-fabricated concentration of approximately 51 at% to 66 at% while U decreased from approximately 21 at% to 15 at%. Neptunium and Pu elemental trends tended to follow those of U, while Am, excluding that occurring in minor precipitates, tended to behave similarly to Zr. Beginning in the fuel center and extending toward the radius approximately 1 mm, the fuel was a relatively homogeneous phase; however, beyond this single-phase region, the fuel separated into two primary phases— a (U, Np, Pu) Zr<sub>2</sub> – like phase and a high uranium content- low zirconium content phase. Americium, lanthanide elements and actinide elements formed multiple lenticular-shaped Nd<sub>7</sub>(Pd, Rh)<sub>3</sub> – like phase precipitates located approximately mid-radius. Fuel-cladding interactions were minor, with Sm and Am penetrating up to 15  $\mu\text{m}$  into the cladding along presumed grain boundaries. These analyses demonstrate the importance and value of using EPMA to characterize irradiated nuclear fuel.