Determination of Free Hydroxide and Aluminum Concentrations by Acid/Base Titrations

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A new analytical method to determine the concentrations of free hydroxide and aluminum in a hot cell environment has been established. This method has the advantage of providing both free hydroxide and aluminum concentrations through simple reverse acid titrations, thus eliminating the need for radioactive sample transfer and handling. Aluminum-curium or aluminum-neptunium cermet pellets assembled in targets are irradiated in the High Flux Isotope Reactor to produce californium-252 or plutonium-238 respectively. The first processing step for the recovery of these isotopes is the caustic dissolution of the aluminum cladding of the target rod and of the aluminum present in the pellets. Until recently, the caustic solution containing the dissolved aluminum was submitted for analysis to quantify aluminum concentrations via mass spectrometry involving contact handling of highly radioactive solutions containing fission products and multiple dilutions to account for sample activity and aluminum concentrations. The free hydroxide was determined via potentiometric reverse titration inside the hot cell. Careful examination of the titration curve revealed that aluminum concentrations could be calculated using selected endpoints, saving time and reducing error associated with performing serial dilutions, and reducing exposure risks by remaining entirely in the hot cell environment.

To prove the validity of these results, we initiated a non-rad study and titrated a series of solutions that were varied systematically in concentrations of sodium hydroxide, sodium nitrate, and aluminum. Direct and reverse titrations were performed, and the analysis of the results led to the validation of this technique along with its limits, allowing operators to perform more rapid direct analysis in support of national plutonium and californium production goals.

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