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Quantification of plutonium by X-ray fluorescence spectroscopy on solid deposit samples from UP1 plant in CEA Marcoule

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1. Introduction

In recent years, various characterizations have been carried out in the Atalante facility in Marcoule on deposits coming from fission product tanks located in the high level activity area of UP1 plant. Among these characterizations, the plutonium concentration is an essential data in order to better control the criticality safety of future recovery operations of these irradiating solid deposits. To have access to this concentration, specific dissolutions in nitrohydrofluoric medium must be implemented in hot cells, which is often a delicate and time-consuming process.

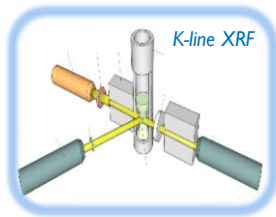
In order to limit the need for destructive analyses, studies have recently been conducted to quantify the plutonium directly into solid deposit samples by X-ray fluorescence spectroscopy.

2. R&D approach and results obtained

K-line X-ray fluorescence is the most appropriate technique for this study. Indeed, this technique, implanted in a hot cell in the Atalante facility, is a direct, fast and non-destructive technique and has the advantage of being less sensitive to solid heterogeneity and matrix effects than low energy XRF.

This technique is commonly used in Atalante facility to determine high concentrations of plutonium in liquid samples. For this study, the measurement geometry had to be modified to be able to specifically analyse solid samples, and a new calibration was performed. The second step consisted in adapting the theoretical model classically used for liquid samples. This new model was finally validated on real solid samples, before and after dissolution.

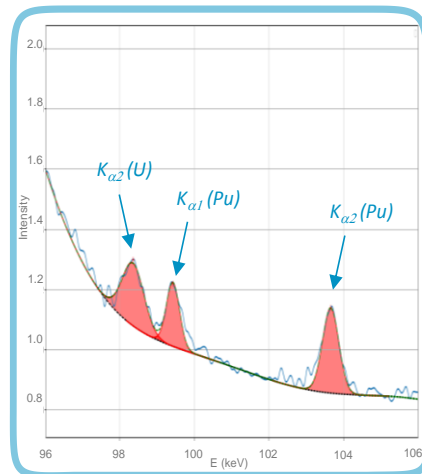
Choice of analytical technique



Measurement geometry optimization



Adaptation of the theoretical model



Validation on real samples



Figure 1: Illustration of the different phases of the study

The plutonium concentrations calculated using the theoretical model on solid samples were compared to the concentrations obtained after dissolution: discrepancies of less than 30% were obtained (the lower limit was estimated at 0.3 mg.g^{-1} of Pu).

3. Conclusion

Direct solid-state analysis by K-line X-ray fluorescence helps determining the plutonium concentration within 30% in highly irradiating deposit samples, without prior dissolution (significant time saver).

This method is independent of isotopic variations and can be applicable to other elements, such as uranium, provided the concentrations are significant in the solid sample. Another major interest is to carry out solid sampling studies (investigation of deposits homogeneity) not only based on gamma emitters but also on alpha emitters.

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