Accuracy and Detection Limit Validationfor Fluorine by INAA at HANARO

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1. Introduction

Instrumental Neutron Activation Analysis (INAA) is a reliable and non-destructive technique for multi-element quantification.[1] However, elements forming short-lived radionuclides remain analytically challenging due to rapid decay during irradiation–transfer–counting sequences. Fluorine is activated via the $^{19}F(n,\gamma)^{20}F$ reaction, producing ^{20}F ($T_{½}$ ≈ 11 s) with a characteristic 1633.6 keV gamma-ray (fig.1). The short half-life requires strict optimization of irradiation time, pneumatic transfer delay, and counting duration.

This study establishes a metrologically validated protocol for fluorine quantification using NIST SRM 180 (CaF_2) at the HANARO research reactor. Quantitative accuracy, precision, detection limits (LOD), sodium interference effects, and uncertainty budget were systematically evaluated.

2. Methods and Results

2.1 Irradiation and Measurement Conditions

Irradiations were conducted at 27 MW reactor power using PTS#1 (NAA#1 hole) with an average thermal neutron flux of approximately 4.8×10^{13} $n \cdot cm^{-2} \cdot s^{-1}$. Irradiation times were varied between 5–30 s to optimize signal-to-background ratio. Cooling/transfer times were controlled within 2–10 s, and counting times were varied between 10–30 s.

Gamma-ray spectra were measured using a 40% relative efficiency HPGe detector. Absolute efficiency calibration (59–1836 keV) was fitted using a polynomial function. Decay and dead-time corrections were applied to all measurements.

2.2 Calibration and Linearity Evaluation

Calibration curves were constructed using SRM 180 mass steps (5, 10, 20, 40, 80 mg). For each mass, five repeated measurements were performed. Net peak areas of the 1633.6 keV gamma line (were plotted against fluorine mass, and linear regression analysis was conducted to evaluate linearity (R^2) and residual distribution.

2.3 Sodium Interference Assessment

To evaluate sodium interference, SRM 180 samples were measured alone and with Na_2CO_3 or NaCl added at multiple concentration levels. The ^{24}Na indicator peak was used to establish a correction model. The influence of Na on fluorine peak area and accuracy was quantitatively analyzed before and after correction.

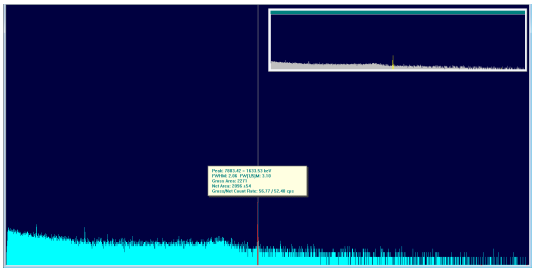

Fig. 1. Gamma-ray spectrum of of SRM180.

2.4 Performance Evaluation

Quantitative accuracy was assessed via recovery (%) and bias relative to certified SRM values. Precision was evaluated using relative standard deviation (RSD%) from repeated measurements. Detection limits (LOD) were calculated according to Currie's method using background count statistics. An uncertainty budget incorporating counting statistics, mass measurement, and transfer delay was established.

3. Conclusions

A comprehensive validation of fluorine quantification using short-irradiation INAA at HANARO has been achieved. The study establishes optimized irradiation parameters, demonstrates validated quantitative accuracy and detection limits, and provides a sodium interference correction model. The developed methodology enhances the metrological reliability of fluorine analysis and supports applications in PFAS-related environmental monitoring.

REFERENCES

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