

# Experimental report

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**Proposal:** 3-17-65

**Council:** 10/2022

**Title:** Broadening the dynamic range of PGAA using high-efficiency detector array

**Research area:** Nuclear and Particle Physics

**This proposal is a new proposal**

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**Samples:** Fe

Instrument	Requested days	Allocated days	From	To
FIPPS	7	7	26/06/2023	03/07/2023

## Abstract:

We want to study the improvement we can achieve in the analytical sensitivity of prompt gamma activation analysis using the most advanced gamma-spectrometry system. The high counting efficiency and the coincidence possibility with multiple HPGe detectors offers a unique opportunity to unfold the spectra where a strong matrix masks the weak signal from trace components, like Cl a highly important corrosive in Fe or silicate matrices. The detection limits and the dynamic range for such measurements have already been determined at FRM II, Garching in high-flux cold neutron beam with a single Compton-suppressed HPGe detector and were found to be about 80 ppm for the iron matrix. More than one order of magnitude improvement is expected. Small-mass model samples will be used with known amount of trace Cl on the 1-100 ppm level using Fe (and Si) plates/wafer together with PVC films. The results will help us to conclude about the development strategies in nuclear analytics.

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Broadening the dynamic range of PGAA using high-efficiency detector array

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We performed the first series of measurement at the FIPPS facility to characterize it from the point of view of high-sensitivity nuclear analytical chemistry. There are a few well-known examples in the analytical routine where limitations of detecting certain elements in the matrix of others were observed at standard prompt gamma activation analysis facilities. One of them is the detection of the traces of the important corrosive element chlorine in archeological iron objects or even cement and concrete. Another famous and important case is that of the oxygen, which normally is the dominant component in natural samples and in most cases, we cannot analyze it with reasonable accuracy because its weak peaks are hidden in the spectral background even in the case of a matrix of medium-cross-section elements.

### Experimental

In this first series of experiment, our goal was to characterize the FIPPS facility and acquire spectra of samples representing the mentioned crucial cases. The characterization meant the precise efficiency calibration with the typical radioactive sources and nuclear reactions used for high-energy detector calibrations: Eu-152, chlorine and nitrogen neutron capture with samples of PVC and Urea-D. Background measurements were also performed to check for chlorine background.

Then a series of samples were prepared from iron sheets about 300 mg each and small PVC pieces with the masses between 0.3 and 23 mg. Measurements were then made with highly different Cl/Fe ratios down to about 100ppm Cl content and pure iron. The spectra were then taken for several hours. Normal spectra (i.e. the sum of individual detectors) were collected and coincidence projections were also generated.

Urea-D also contained stoichiometric amount of oxygen, where the coincidence possibilities of oxygen measurements could also be checked quickly.

### Results

It was observed that Cl appears in the beam background, thus, the lowest concentration regions could not be reached in the present series of experiments. This is illustrated in the next Figure.

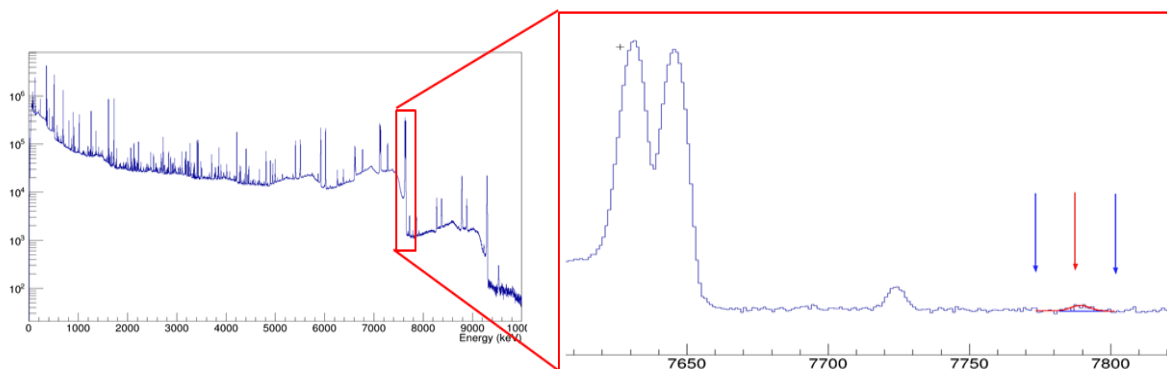


Figure 1. The normal spectrum of pure iron. In the magnified region, the characteristic doublet of Fe is displayed at the energies 7631 and 7645 keVs. A characteristic line of Cl is marked at 7790 keV.

The peak area in the iron doublet at 7631+7645 keVs was found to be  $2.27 \times 10^6 \pm 0.1\%$  while the chlorine line at 7790 keV reached the area of 1540 with 5% uncertainty. From this, the detection limit (i.e. the 50-% quantification limit) is estimated to be  $\sim 150$ . Converting this to mass values using the efficiency, it means 15 ppm Cl in Fe matrix. In this model experiment, we disregarded that the Cl signal partly originates from the background producing alone a signal equivalent to about 150 ppm in this iron quantity. The beam intensity had to be attenuated for these experiments.

The coincidence projection of the same measurement revealed the following, as shown in the next Figure.

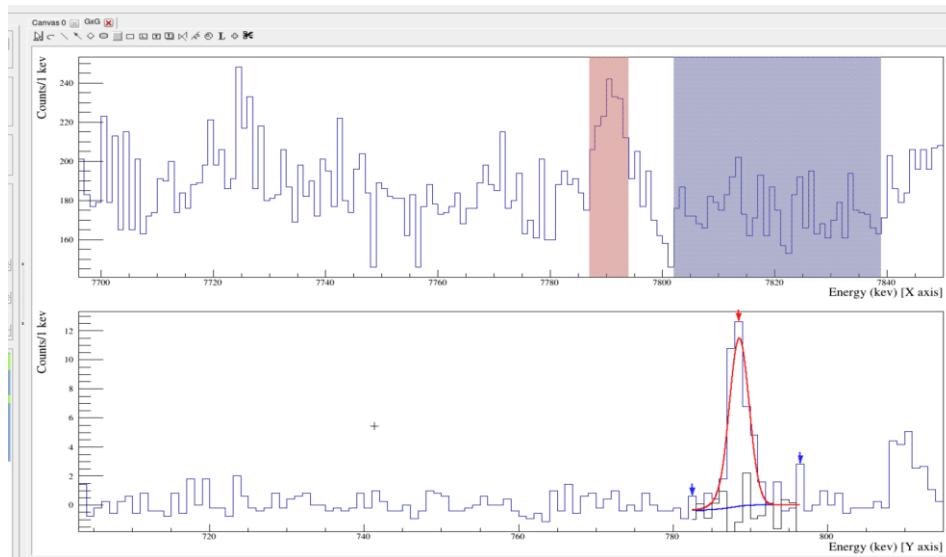


Figure 2. coincidence between the chlorine lines.

A well-known double cascade from Cl-36 can be observed in coincidence mode much better: the two energies are 7790 keV and 788 keV. As can be seen, the low-energy peak (below) in coincidence with the high-energy peak (above), becomes clearly distinguishable, while it remains invisible in the normal spectrum.

The coincidence method was also tested on the spectrum of urea, where the lines of oxygen could be found in a similar way.

Measurements of silicate samples could not be performed in this campaign.

## Conclusions

Using multiple detectors increases the sensitivity of the analysis significantly. It has been also shown that the coincidence mode can be used for finding hidden characteristic peaks. The quantification still needs to be developed as the determined mass values in this mode are dependent not just on the partial gamma-ray production cross sections, but also on the branching ratios. Further interpretation for the data is needed, especially regarding the quantification of coincidence spectra. Further experiments will be needed important light elements, first of all oxygen and the chlorine content of silicate samples. In the future measurements, the new facility needs to be characterized again, especially regarding the chlorine background. A special care must be taken at the beam attenuation to get reproducible flux values and homogeneous beam cross sections.