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STUDY AND CALIBRATION OF THE HPGE DETECTOR FOR RADIONUCLIDE ANALYSIS OF IODINE-125

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ABSTRACT

In this work, a theoretical-experimental methodology was developed for the calibration of HPGe Detector for quantitative analysis of iodine-125. Photopick efficiencies was measured between 0 - 40 keV using a certified calibration source (iodine-129) with well-known activity. The efficiency curve was determined for an energy range that covers the region of interest for iodine-125 (0 - 40 keV). The calibrate spectrum was obtained for study and the result was saved in the detector directory for future analysis and determination of the iodine-125 activity.

1. INTRODUCTION

1.1. HPGe Detector

High Pure Germanium detectors (HPGe) are composed of semiconductor diodes having a p-i-n structure in which the intrinsic region (i) is sensitive to ionizing radiation, particularly x-rays and gamma rays. When the photons interact with the material within the confined volume of a detector, the charge carriers (electrons) are produced and are swept by the electric field to the electrodes p and n. This charge, which is proportional to the energy deposited in the detector by the input photon, is converted into a voltage pulse by an integral load-sensitive preamplifier. An analyzer discriminates the pulses, which are released in a gamma spectrum at the software interface. The program provides the number of decays for any radioactive material, which are used to calculate the activity [1]. It is necessary to pre-calibrate the equipment to determine its efficiency, which is the purpose of this work.

1.2. Energy Calibration

The object of the energy calibration is the relationship between the position of the peak in the spectrum channel and its corresponding gamma rays. Calibration is done by measuring the spectrum of a source that emits gamma rays with a precise energy and comparing it at the peak energy position. It does not matter if the source has one or many nuclides. Regardless of the source used, it is advisable to ensure that the calibration energies cover the entire range over which the spectrometer will be used. In practice, it is necessary to measure the spectrum long enough to achieve good statistical accuracy for the peaks used in the calibration. Normally two or more points are used to adjust a straight line of energy calibration. Approximate uncertainties for a range of 0 to 2000 keV are about 1 keV, and increases at the ends of the energy range. Extrapolations beyond the calibrated points can lead to uncertainties too [2].

1.3. Peak Width Calibration

If a spectrometer is used simply with peak areas derived from regions of interest defined manually, there is no need to perform peak width calibration. The peak width depends on several statistical uncertainties of the production process, collecting and transmitting signals to the system [2]. The peak width is represented by 3 factors:

- Uncertainty of the number of electron pairs created by the gamma interaction;
- Uncertainty of the collected charge;
- Electronic noise.

To estimate the peak width, the following equation is used, which approximates the peak of a Gaussian distribution:

$$FWHM = 0.939\frac{A}{C_T - C_0} \tag{1}$$

Where FWHM (full width half maximum) is the peak width at half height, A is peak area, C_T is the total height of the peak, C_0 is the height of the background. Fig. 1 illustrates these specifications.

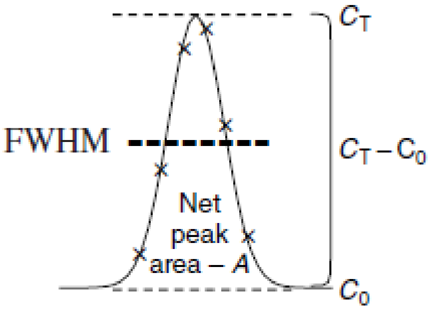


Figure 1: Specification of the values used to calculate the peak width [2].

1.4. Absolute Full-Energy Peak Efficiency

The intent of gamma spectrometry is to relate the peak area in the spectrum to the amount of radioactivity it represents. For this it is necessary to know the absolute full-energy peak efficiency [2]. This relates the area of the peak to the number of gamma rays emitted by the source and should depend on the geometric arrangement of the source and the detector. Total peak efficiency is the parameter of greatest significance in gamma spectrometry. The calculation is straightforward; it is the ratio of the number of counts detected in a peak to the number of counts emitted by the source:

$$\varepsilon = \frac{C - C_0}{S.P_\gamma.t} \tag{2}$$

C: Counts throughout the area of a peak subtracted from its respective background (C_0).
S: Source activity in Bq, corrected by the present time since date calibration.
P_γ: Probability of emission of the particular gamma-ray being measured
t: count time.

1.5. Uncertainty Calculation

For a set of n measurements, the experimental standard deviation is a parameter that characterizes how scattered the obtained values are. This means that if the results are very close to each other than the standard deviation will be “small” and if the results are scattered the standard deviation will be “large”. The experimental standard deviation (s) can be calculated by the variance (s^2):

$$s^2 = \frac{1}{n-1}\sum_{i=1}^{n}(y_i - \bar{y})^2 \quad (3)$$

$$s = \sqrt{\frac{1}{n-1}\sum_{i=1}^{n}(y_i - \bar{y})^2} \quad (4)$$

To know the mean value, infinite measurements would have to be made. Since this is not possible in practice, we know that the mean is just an estimate of the true mean value. In this context, the standard deviation of the mean value is a parameter that tells how well the mean of observations is an estimate of the true mean value. Qualitatively, “small” standard deviation values from the mean value are associated with good estimates of the true mean value; on the contrary, high values represent that the mean is not a good estimate of the true mean value. The standard deviation of the mean value (s_m) is calculated by:

$$s_m = \sqrt{\frac{1}{n(n-1)}\sum_{i=1}^{n}(y_i - \bar{y})^2} = \frac{s}{\sqrt{n}} \quad (5)$$

2. METODOLOGY

The HPGe detector used was the ORTEC, model GEM-C5970-B connected in a liquid nitrogen reservoir (LN2 Dewar). This detector was fully configured for the study in question. Since the energies of the calibration spectrum are concentrated in a region of lower energy, the gain of the amplifier was adjusted at 5.60 (0.7 for fine gain and 8 for course gain) and worked with 4096 channels of resolution; all this in order to transfer the peaks to the middle of the spectrum, to place them in a region of higher resolution. The detector was operated at 3500V. The calibration source chosen was a point source of iodine-129, since its energy peaks cover the entire iodine-125 energy range.

Figure 2: Point source of iodine-129.

Initially the analysis was performed without the presence of the emitting source, in order to measure the background radiation; the spectrum was collected and stored to correct the real spectrum of the source. In Annex A it is possible to see the values collected.

In the second step, the source was inserted into the detector and its energy peaks were measured for the calibration of the system, also during a period of 1 hour. The source was fitted in a sample holder coupled to the HPGe detector window to ensure that the analysis was always performed in the same source/detector position. Fig. 3 illustrates this configuration. The sample holder was designed in low-density material to attenuate the x-radiation emitted by the source in order to generate a cleaner spectrum with less interference.

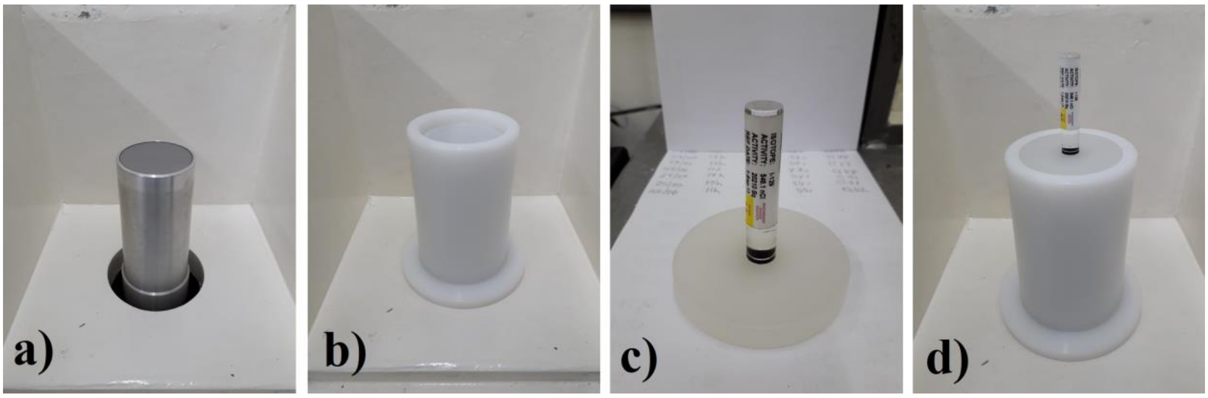


Figure 3: Assembly of the sample holder for measuring the source always in the same position. a) Detector window; b) sample holder placed; c) moving part of the sample holder; d) calibration source being analyzed.

The analysis was repeated 3 times for collection of 3 comparative spectra. With the obtained data it was possible to calculate the variance, standard deviation and average standard deviation of the efficiencies of the three spectra.

3. RESULTS

Starting with the background collection the following spectrum was generated (Fig. 4):

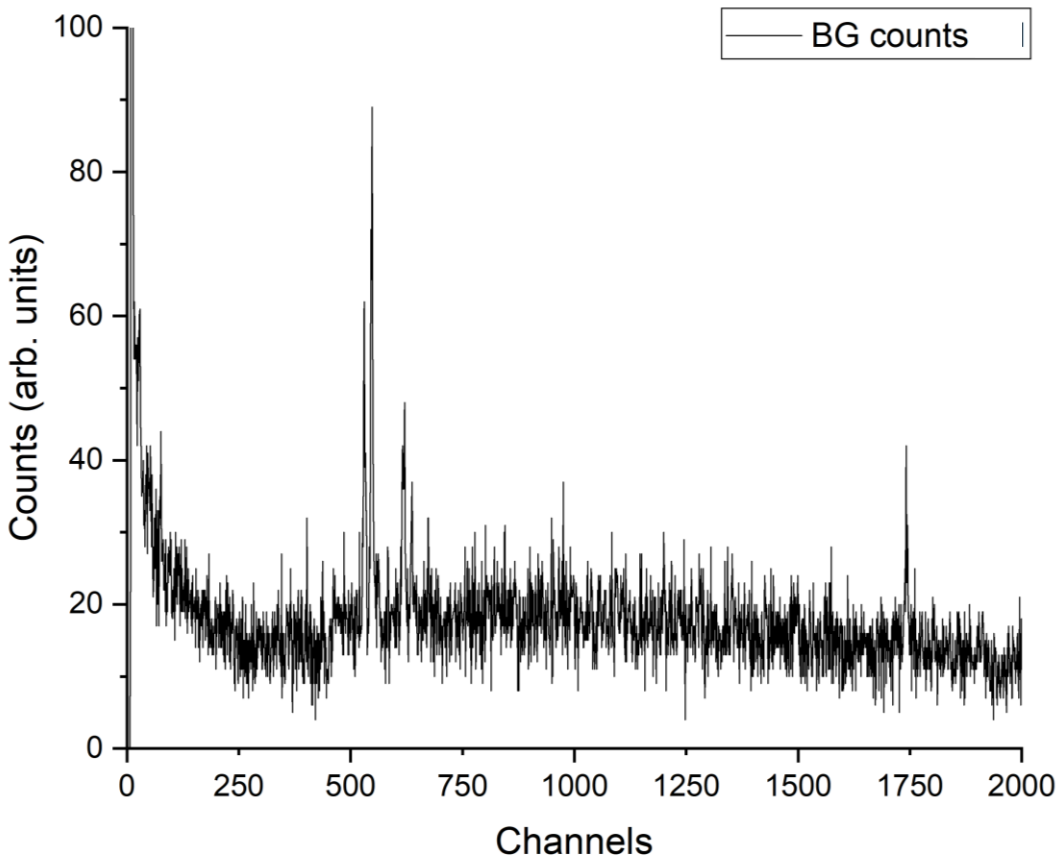


Figure 4: Spectrum without the presence of emitting source.

For the calibration of energy, iodine-129 was measured for 1 hour to guarantee photopics with more than 100000 counts generating the following spectrum (Fig. 5):

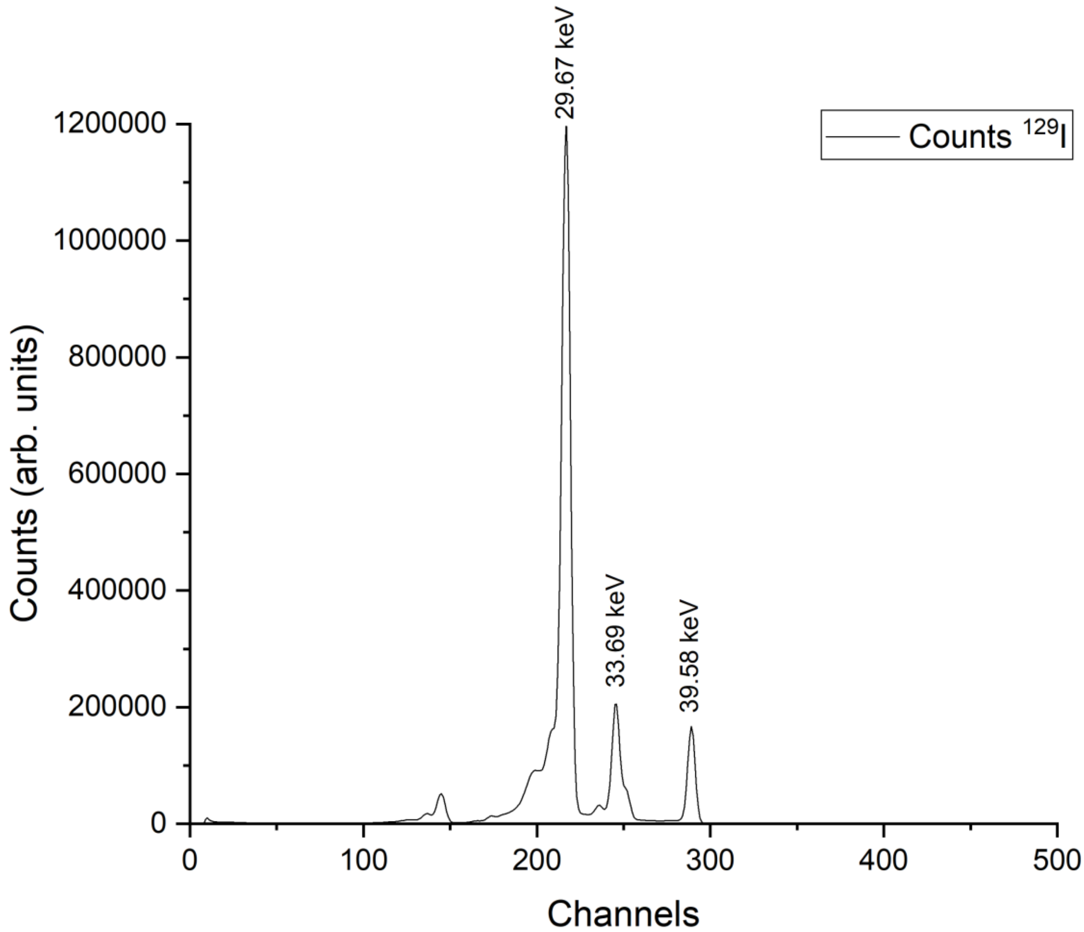


Figure 5: Spectrum of iodine-129 energy with its characteristic peaks.

The peaks are found in the literature and represent the following energies represented in the Table 1:

Table 1: Characteristics peaks of iodine-129.

Energy (keV)	Intensity (%)
29.667	57.3
33.689	10.30
39.578	7.42

Source: NUCLEIDE, L, 2019 [4].

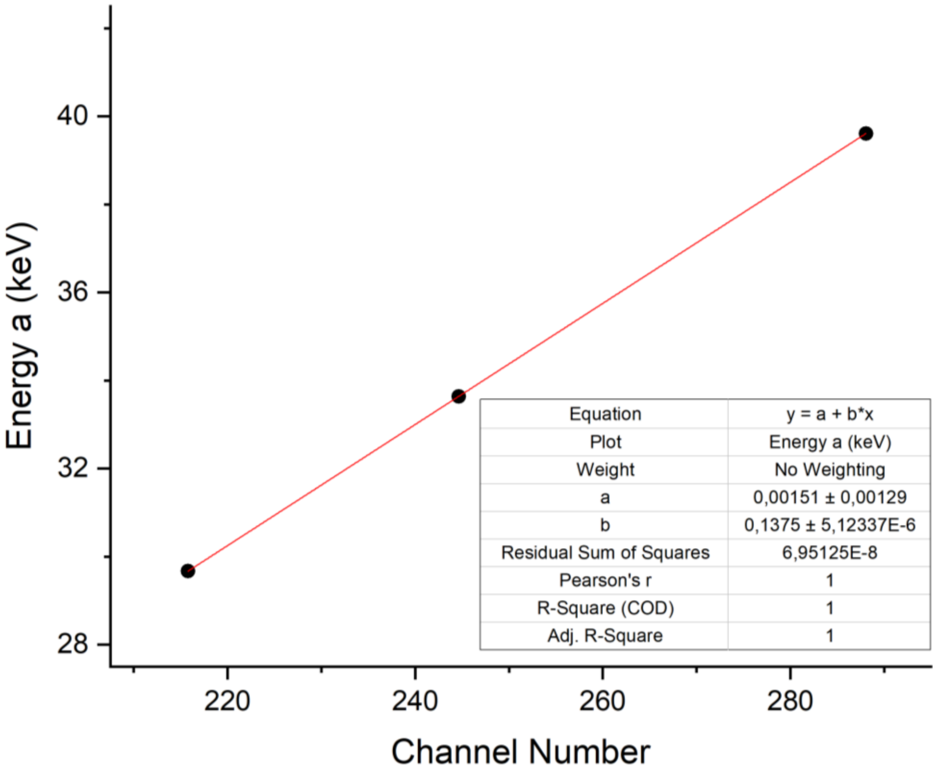
These values were entered into the detector channels according to the Table 2:

Table 2: Energy peaks in respective channels.

Channel Number	Energy [a] (keV)
215.77	29.67
244.64	33.64
288.06	39.61

a. The energies undergo a small change when inserted in discrete channels.

They are used in energy calibration, resulting in the following energy chart per channel (Graph 1):



Graph 1: Energy calibration fit.

An excellent adjustment between the points and a calibration with high accuracy was observed.

For peak width calibration, the MAESTRO software [3] has an internal algorithm that automates the width calculation, so no manual calibration is required.

For the calculation of absolute efficiency, the information that satisfies equation 2 (gross area counts, background counts, corrected activity, probability of emission and time) was collected. The efficiency of each peak was calculated according to the following tables (Table 3, 4 and 5):

Table 3: Spectrum information 1.

Spectrum 1							
Channel	Energy (keV)	Gross Area Counts	Background Counts	Time (s)	Corrected Activity (Bq)	Probability of Photon Emission	Absolute Energy Peak Efficiency
215.77	29.67	8704492	397	3600	20209.99	0.573	0.2088
244.64	33.64	1505751	232			0.103	0.2009
288.06	39.61	925172	353			0.074	0.1713

Table 4: Spectrum information 2.

Spectrum 2							
Channel	Energy (keV)	Gross Area Counts	Background Counts	Time (s)	Corrected Activity (Bq)	Probability of Photon Emission	Absolute Energy Peak Efficiency
215.79	29.67	8712993	397	3600	20209.99	0.573	0.2090
244.65	33.64	1505104	232			0.103	0.2008
288.07	39.61	925586	353			0.074	0.1714

Table 5: Spectrum information 3.

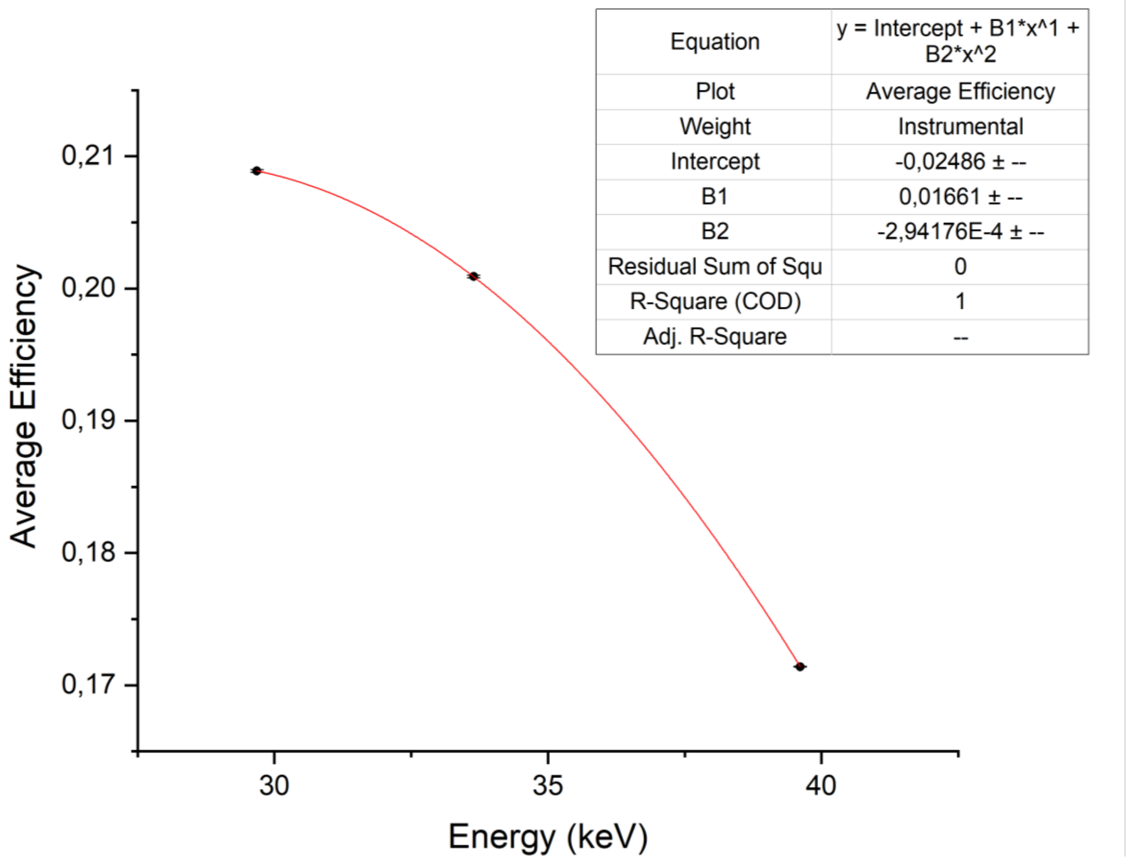
Spectrum 3							
Channel	Energy (keV)	Gross Area Counts	Background Counts	Times (s)	Corrected Activity (Bq)	Probability of Photon Emission	Absolute Energy Peak Efficiency
215.84	29.68	8716317	397	3600	20209.99	0.573	0.2091
244.67	33.64	1507379	232			0.103	0.2011
288.08	39.61	925700	353			0.074	0.1714

After calculating the efficiencies, the average efficiencies of the coincident peaks in relation to the 3 spectra were calculated, as well as their variances, standard deviations and average standard deviations.

Table 6: Average Efficiency, variance, standard deviation and average standard deviation respectively.

Energy (keV)	Average Efficiency	s^2	s	s_m
29.68	0.2089	2.140E-08	0.0001	0.0001
33.64	0.2009	2.447E-08	0.0002	0.0001
39.61	0.1714	2.649E-09	0.00010	0.00003

These data were used in the construction of the absolute energy efficiency graph (Graph 2).



Graph 2: Efficiency calibration fit.

With the results of this calibration, it is possible to observe that the efficiency decreases with the increase of energy, according to a second-order polynomial function. This relationship is common and also indicated by the literature. The fit is optimal, and the correlation between efficiency/energy is consistent. These graphics and equations can be used for the sample activity analysis.

3. CONCLUSIONS

The high pure germanium detector was calibrated in energy and efficiency following the instructions of literature and showed good reproducibility with low data dispersion and acceptable adjustments. The system is ready to receive the iodine-125 samples that will be produced in the IEA-R1 nuclear reactor. The calculation of the activity of these samples of iodine-125 will be part of a work that will be done later because it depends on several factors such as geometry and density.

ACKNOWLEDGMENTS

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ANNEX A

```
Detector #1      ACQ 25-jun-2019 at 14:34:02   RT = 4168.0   LT = 3600.0
          LABELISA-NET1 DSPEC-JR2 SN 17108232
          129i-3600s-25/06/19-1corrida

ROI # 1     RANGE: 202 = 27.77keV  to  225 = 30.94keV
          AREA : Gross =  8704492    Net = 7244196   +/- 3409
          CENTROID: 215.77  = 29.67keV
          SHAPE: FWHM = 0.78     FW(1/5)M = 1.19
          ID: I-129 at 29.78keV

ROI # 2     RANGE: 238 = 32.72keV  to  255 = 35.06keV
          AREA : Gross =  1505751    Net = 1047192   +/- 1401
          CENTROID: 244.64  = 33.64keV
          SHAPE: FWHM = 0.68     FW(1/5)M = 1.30
          ID: I-129 at 33.60keV

ROI # 3     RANGE: 276 = 37.95keV  to  300 = 41.25keV
          AREA : Gross =  925172    Net = 855905   +/- 1036
          CENTROID: 288.06  = 39.61keV
          SHAPE: FWHM = 0.68     FW(1/5)M = 1.04
          ID: I-129 at 39.52keV
```

```
Detector #1      ACQ 25-jun-2019 at 16:12:17   RT = 4169.5   LT = 3600.0
          LABELISA-NET1 DSPEC-JR2 SN 17108232
          129i-3600s-25/06/19-2corrida

ROI # 1     RANGE: 202 = 27.77keV  to  225 = 30.94keV
          AREA : Gross =  8712993    Net = 7255525   +/- 3409
          CENTROID: 215.79  = 29.67keV
          SHAPE: FWHM = 0.78     FW(1/5)M = 1.19
          ID: I-129 at 29.78keV

ROI # 2     RANGE: 238 = 32.72keV  to  255 = 35.06keV
          AREA : Gross =  1505104    Net = 1047400   +/- 1401
          CENTROID: 244.65  = 33.64keV
          SHAPE: FWHM = 0.67     FW(1/5)M = 1.29
          ID: I-129 at 33.60keV

ROI # 3     RANGE: 276 = 37.95keV  to  300 = 41.25keV
          AREA : Gross =  925586    Net = 857286   +/- 1036
          CENTROID: 288.07  = 39.61keV
          SHAPE: FWHM = 0.68     FW(1/5)M = 1.04
          ID: I-129 at 39.52keV
```

Detector #1   ACQ 25-jun-2019 at 17:23:54   RT = 4171.0   LT = 3600.0
LABELISA-NET1 DSPEC-JR2 SN 17108232
129i-3600s-25/06/19-3corrida

ROI # 1   RANGE: 202 = 27.77keV to 225 = 30.94keV
AREA : Gross = 8716317   Net = 7256241   +/- 3411
CENTROID: 215.84 = 29.68keV
SHAPE: FWHM = 0.77   FW(1/5)M = 1.19
ID: I-129 at 29.78keV

ROI # 2   RANGE: 238 = 32.72keV to 255 = 35.06keV
AREA : Gross = 1507379   Net = 1050761   +/- 1401
CENTROID: 244.67 = 33.64keV
SHAPE: FWHM = 0.67   FW(1/5)M = 1.28
ID: I-129 at 33.60keV

ROI # 3   RANGE: 276 = 37.95keV to 300 = 41.25keV
AREA : Gross = 925700   Net = 856283   +/- 1037
CENTROID: 288.08 = 39.61keV
SHAPE: FWHM = 0.67   FW(1/5)M = 1.03
ID: I-129 at 39.52keV