



Potassium-40 determination in NPK fertilizer by CZT-500s gamma spectrometry in the LPNPE Lab

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Abstract: The isotope 40 of Potassium (^{40}K) is determined in NPK fertilizer by gamma spectrometry in the LPNPE Lab. For radioactive nuclide identification, the gamma spectrometry method with a CZT-500s detector is utilized and ^{137}Cs coupled with ^{131}I sources are used for energy calibration. The ^{40}K identification is carried out by sampling of 500ml in a marinelli type beaker and based on the photopeak of 1460.8 keV. The environmental background is suppressed by applying shielding technique. The shielding is made of 8cm thick walls of lead and reduces the natural background of around 20 times. The peak position of ^{40}K has been precisely determined on the energy regions from 50keV to 2120keV.

Keywords: ^{40}K , Gamma spectrometry, CZT-500s

I. Introduction

Mineral and natural resources usually contains long half-life natural radioactive nuclides (such as ^{238}U , ^{232}Th and ^{40}K) and had been greatly exploited for different utilizations.

In the LPNPE Lab, ^{40}K is determined in NPK fertilizer by gamma spectrometry. The objectives are firstly to calibrate the multichannel analyzer and secondly use it to acquire gamma-ray spectra, and to locate primary photopeaks. The spectra are measured using the CZT-500s detector coupled with the MCA-166 and lead shielding method is applied for background subtraction. Indeed, many users of gamma spectrometer are concerned with measuring low levels of radioactivity such as found in routine environmental samples. For these, the background should be as low as possible to provide lower detection limit. In the present work, a successful approach for this study is to suppress the Background by Special Detector Shielding Materials (Lead).

The background radiation is mainly due to a combination of:

- Environmental terrestrial gamma radiation and laboratory structures,
- Radioactivity in air surrounding the detector: mainly due to radon gas,
- Primary and secondary components of the cosmic radiation.

Natural potassium comprises three isotopes (^{39}K , ^{40}K , ^{41}K) where ^{40}K is the only radioactive potassium isotope having a natural isotopic abundance of 0.0118%. The beta and electron capture decay modes of ^{40}K to ^{40}Ca (89.28%) and ^{40}Ar (10.72%) respectively (the latter followed by the emission of a 1460.8 keV gamma ray) contribute significantly to the natural radioactivity.

Potassium in rocks is concentrated mainly in potassium feldspars and micas. Potassium is soluble under most conditions and during wet weather is lost into solution. The ^{40}K determination in NPK fertilizer by CZT-500s gamma spectrometry is one method for a preliminary analysis of such environmental minerals in the LPNPE Lab.

II. Energy calibration

The energy calibration converts the x-axis (Channel) number into energy expressed in keV. Many formula exist for energy calibration but in the present work the linear formula have been taken: $E(\text{keV})=a(\text{keV/Ch})\cdot\text{channel} + b(\text{keV})$.

As the available sources at the LPNPE Lab are only I-131 and Cs-137, the energy calibration method is based on these sources' spectra.

As reference for gamma energy emission, the data from the report of the French atomic energy commission "Commissariat à l'Energie Atomique" (CEA) year 2008 (CEA-R-6201) have been taken. From this document, the most important gamma energies emitted by these two elements are listed below:

Cs-137: 661.657keV, emission percentage: 84.99%.

I-131: 284.305keV, emission percentage: 6.06%

364.489keV, emission percentage: 81.2%

636.989keV, emission percentage: 7.26%

The following figure shows the setup relating to the energy full range of about 2MeV (energy regions from 50keV to 2120keV). 2MeV is chosen in order to be able to localise the 1.46MeV peak of ^{40}K in a spectrum. Five spectra series are measured for each source and mean values are used for energy calibration.

Σ Setup summary

MCA Channels: 2048 Energy threshold (%): 2 LLD: 0 ULD: 1983 ADC input: Amplifier		Detector high voltage High voltage (Volt): +1400 HV inhibit mode: unused	
Amplifier Coarse gain: 200 Fine gain: 0.5000 Shaping time (μsec): low Input polarity: pos Pile up rejection: on PZC: 1600		Preamplifier power supply +12 V: on +24 V: on -12 V: on -24 V: on	
		Stabilization Stabilization off	
		Presets Choice: Live Time (sec) Value: 300 ROI	

Fig. 1 Setup summary for energy calibration

Five spectra of I-131 are measured and the mean values as well as the standard deviations of peak centroid positions are shown in the following table.

	channel centroid ROI=248-278	channel centroid ROI=296-350	channel centroid ROI=586-604
Mean values	265.1	339.4	593.9
Standard deviation	0.4	0.2	0.5

Table 1. Identification of I-131 peaks positions for energy calibration

The same spectra measurement process for Cs-137: Five spectra are measured and the mean values as well as the standard deviations of peak centroid positions are shown in the table below.

	channel centroid ROI=584-634
Mean values	616.6
Standard deviation	0.6

Table 2. Identification of Cs-137 peak position for energy calibration

The linear fitting curve coefficients a and b as well as the R^2 (a measure of how well the fit function follows the trend in the data.) are obtained by least squares fit method (a small program written and performed at LPNPE Lab is used) and using the mean values of peak centroid positions as follows:

I-131: 284.305keV / 265.1 Ch
 364.489keV / 339.4 Ch
 636.989keV / 593.9 Ch

Cs-137: 661.657keV / 616.6 Ch

This gives:

$a = 1.073 \text{ keV/Ch}$

$b = 0.167 \text{ keV}$

$R^2 = 0.999$

$R^2 \approx 1$ means that the fit function follows the trend of the data. The energy calibration curve is shown in the following figure.

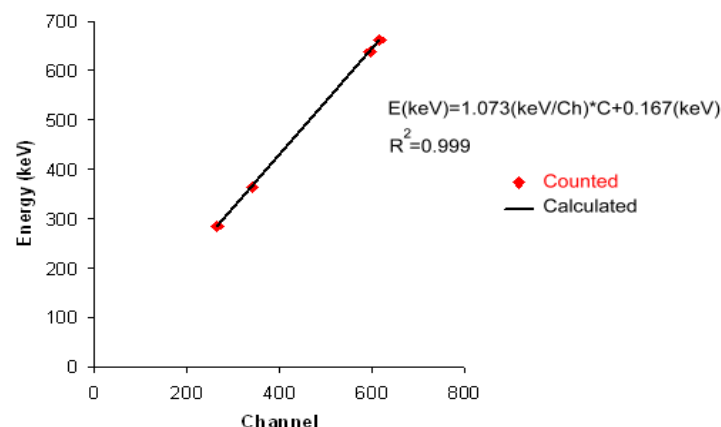


Fig. 2 Energy calibration curve

III. Lead shielding for background subtraction

When gamma radiation of intensity I_0 is incident on an absorber of thickness L , the emerging intensity I transmitted by the absorber is given by the exponential expression $I = I_0 e^{-\mu L}$ where μ is the attenuation coefficient (expressed in mm^{-1}).

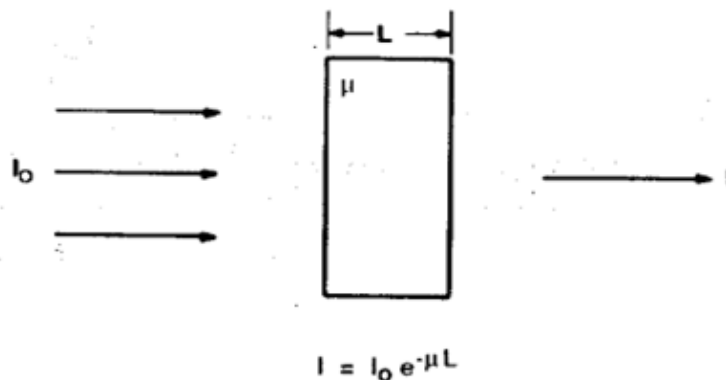


Fig. 3 The fundamental law of gamma ray attenuation. The transmitted gamma ray intensity I is a function of gamma ray energy, absorber composition, and absorber thickness.

Gamma-ray shielding materials should be of high density and high atomic number so that they have a high total linear attenuation coefficient and a high photoelectric absorption probability. The most common shielding material is lead because it is readily available, has a density of 11.35g.cm^{-3} and an atomic number of 82, and is relatively inexpensive. Lead can be moulded into many shapes.

In the LPNPE Lab, lead shielding is conceived and fabricated (Fig.4).

The optimal wall thickness is pre-determined by using the ^{137}Cs source and different thickness of lead (0mm, 5mm, 10mm and 15mm thickness) which are placed between the source and the detector. The attenuation coefficient μ (expressed in mm^{-1}) is determined by the exponential expression $I = I_0 e^{-\mu L}$ and from which the optimal wall thickness is calculated.

The calculation gives:

$$\mu = (0.08 \pm 0.01) \text{mm}^{-1}$$

$$L_{1/10} = (2.8 \pm 0.1) \text{cm} \text{ (thickness of absorber with 10\% transmission).}$$

A lead wall of 8cm thickness is fabricated. This reduces the natural background of around 20 times (of course depending on the energy of photons of gamma radiation) which is optimal for most analysis conducted in the LPNPE gamma spectrometry.

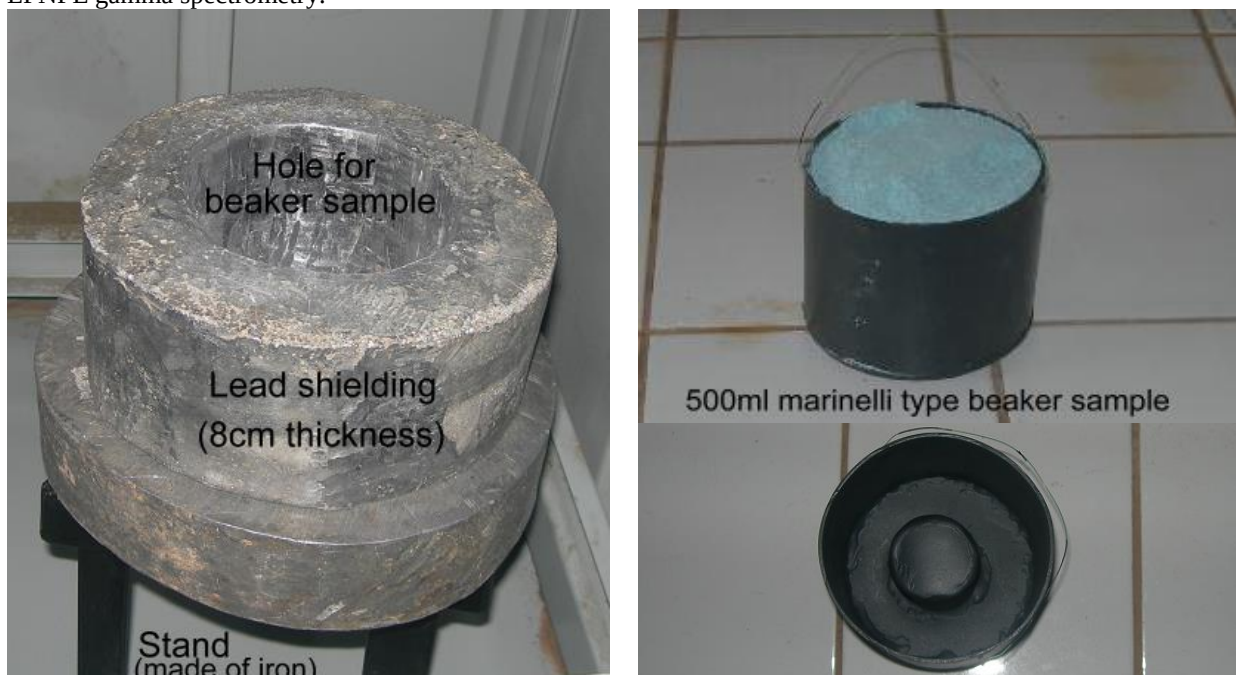


Fig. 4 Lead shielding and marinelli type beaker sample

For sample measurement, the 500ml marinelli type beaker full of sample is put in the hole for beaker sample and the detector is introduced from the bottom of the lead shielding. A 2cm thickness lead cover is afterwards put on top of the beaker.

IV. K-40 determination

Σ Setup summary			
MCA		Detector high voltage	
Channels:	2048	High voltage (Volt):	+ 1400
Energy threshold (%):	2	HV inhibit mode:	unused
LLD:	0	Preamplifier power supply	
ULD:	1983	+12 V:	on
ADC input:	Amplifier	+24 V:	on
Amplifier		-12 V:	on
Coarse gain:	200	-24 V:	on
Fine gain:	0.5000	Stabilization	
Shaping time (μsec):	low	Stabilization off	
Input polarity:	pos	Presets	
Pile up rejection:	on	Choice:	Live Time (sec)
PZC:	1600	Value:	43200
		ROI	

Fig. 5

Data is collected for twelve hours (43200sec) because the amount of ^{40}K in the sample is relatively small. NPK fertilizer's sampling of 500ml has been performed. Five measurements are performed with the setup parameters described in Fig.5 which is the same as the one for energy calibration (Fig.1) except on data collection time (43200sec instead of 300sec). The following figure shows one calibrated spectrum of these series of measurements.

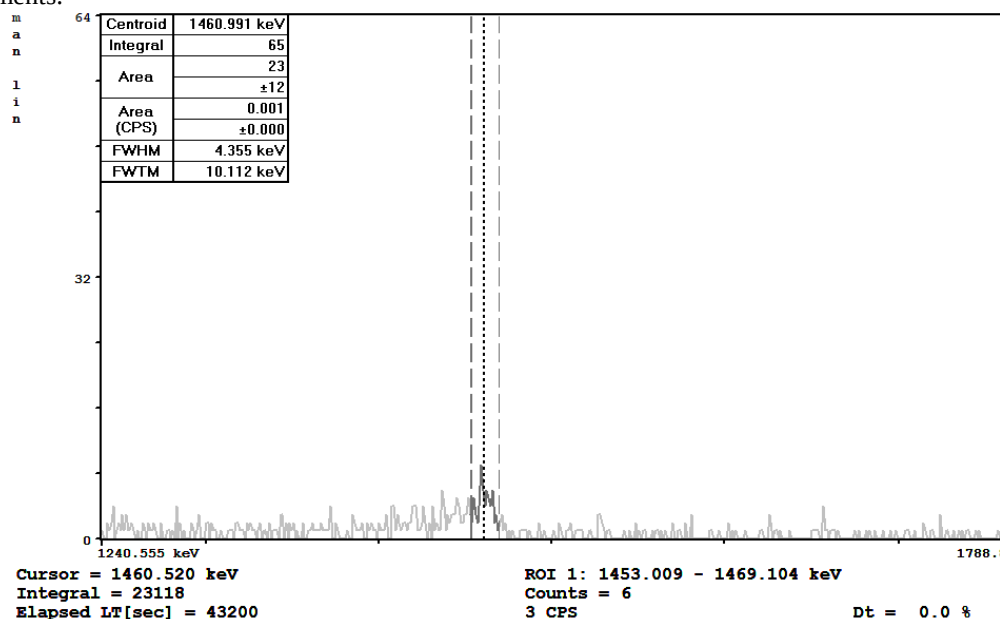


Fig. 6 The 1460.8keV peak of ^{40}K in a NPK fertilizer spectrum

V. Interpretation

All data for radionuclide determination are analyzed in the region between 1453.009keV and 1469.104keV, which encompasses the peak of gamma radiation from ^{40}K . The mean value and standard deviation of peak positions from the five spectra give the peak energy of $(1460 \pm 1)\text{keV}$ which includes 1460.8keV.

The channel containing the peak centroid is used by the software WinSPECi to determine the curve for keV/channel (energy calibration). Calibration result: 1361CH $\rightarrow$ 1460.52keV

The Fig.6 shows one example of ^{40}K peak position: the 1460.52keV peak where the cursor is placed.

VI. Conclusion

The gamma spectrometry method used in the measurement was found to be fairly good to detect the presence of ^{40}K in the NPK fertilizer sample. Indeed, the precision of the energy calibration method is found to be good with the R^2 value quite equal to 1 and the calculated peak energy of $(1460 \pm 1)\text{keV}$ indicate that the precision of the measurement is still within the satisfactory level.

The environmental background is suppressed by applying shielding technique. The shielding is made of 8cm thick walls of lead and reduces the natural background of around 20 times which is optimal for most analysis conducted in the LPNPE gamma spectrometry.

Counting time of 12 hours (43200sec) is found to be sufficient enough to enable the spectrometry system to detect ^{40}K in the NPK fertilizer sample. Overall, this analytical technique is able to produce quality and reliable analytical result for detecting ^{40}K in a sample.

As the LPNPE Lab has not yet a standard source with known activity for efficiency calibration, the procurement of such standard is imperative for the interpretation of spectrum in terms of activity (quantify radionuclide).

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