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Validation of Tritium Calibration Curve in CIEMAT/NIST Activity Measurement Using Non Linear Least Squared Fittings and Calculations of the Half-Life and Decay Constant of Potassium-40

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Abstract

Non-linear curve fitting of tracer efficiency is one of the uncertainty contributors in CIEMAT/NIST method for specific activity measurement and half-life evaluation of radionuclide. This study applied least squared fitting of four different polynomials to validate the tracer efficiency calibration curve for the specific activity measurements of nine Potassium Chloride samples from which half-life and decay constants were computed. All samples were measured using TR1000 Liquid Scintillation Counter and the results of the relative standard uncertainties in tracer interpolated efficiencies associated with the least square fit analysis were found to be 8.363%, 8.076%, 7.941% and 8.767% for polynomials of $n = 2$, $n = 3$, $n = 4$ and $n = 5$, respectively. The corresponding values of ^{40}K specific activity, from the application of empirical efficiencies generated with these polynomials were found to be (16.541, 16.540, 16.537 and 16.548) Bq/g respectively. From these measured specific activity values, the computed half-life and decay constants were found to be (1.2518, 1.2519, 1.2521 and 1.2513) $\times 10^{9}$ y (for $n = 2$, $n = 3$, $n = 4$, and $n = 5$), respectively, and (5.365, 5.5364, 5.5354 and 5.5391) $\times 10^{-10} y^{-1}$ respectively. All values were found to be in good agreement with results of other literatures. However to minimize uncertainty associated with empirically generated tracer interpolation efficiencies, least squared fit analysis of tracer calibration curve should be done, with control trials using different polynomials so as to obtain the best fit.

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Keywords: CIEMAT/NIST method, Liquid Scintillation Counting, Least Square Fit, Analysis, Tracer Calibration Curve

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1. Introduction

Non-linear curve fitting is a very important component in nuclear metrology [1]. In fact, it is applied in analytical formula for half-life evaluation of radionuclide [2, 3] and one of the uncertainty contributors in CIEMAT/NIST method for specific activity measurement [4]. This is because the CIEMAT/NIST method relies on tracer non-linear curve fitting for accurate interpolation of the relative counting efficiency of the radionuclide under investigation [5, 6]. The choice of both Tritium and Potassium-40 (^{40}K) in this study was because of the following reasons: (i) tritium suitability for use in CIEMAT/NIST

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method as a pure beta emitting radionuclide (ii) significance of Potassium-40 (^{40}K) in radiometric age determination [4] which depends solidly on accurately determined half-life and decay constants [7].

^{40}K is a naturally occurring radionuclide aside uranium and thorium [8], whose decay to Calcium-40 (one of the stable isotopes of Calcium) [9, 10] and especially to Argon-40 (figure 1) is widely used in radiometric dating [4]. Unfortunately, uncertainty in half-life and decay constant values have limited present day radiometric dating [4]. More so, the recent attribution of disagreement in the values of decay constants to the method used in determining half-lives by Mc Donough et al, [11] serves as a trigger for this study as slight uncertainty of only one percent in decay constant could lead to significant disagreements in the ages of radioisotopes [7]. Unlike, geochronology, nuclear Physics experiments determine half-life through activity measurement. CIEMAT/NIST efficiency tracing method is one of such methods for specific activity measurement whose worth has been proven in the activity measurements of many radionuclide and employs fitting as the best means of interpolation [4, 5, 6].

In partial response to the call made by half-life evaluators for in-depth assessment of individual uncertainty components for good half-life measurement [12], this paper therefore shows how to validate the tracer (Tritium) calibration curve through controlled trials of non-linear least square fit analysis with different polynomials to determine the relative uncertainty associated with interpolated counting efficiencies of tracer (Tritium). This was achieved with the use of CIEMAT/NIST method. TR 1000 Liquid Scintillation Analyzer was used to measure the specific activity of ^{40}K in Potassium Chloride (KCl) samples from which the half-life and decay constant were evaluated.

2. Materials and Methods

2.1. Sample Preparation and Counting

A total of nine (9) samples were prepared as follows: 15 ml of Ultima-Gold AB Scintillation cocktail was added each to 9 glass vials(20 ml capacity) of low potassium content. In a similar manner, 1 ml of Potassium chloride (KCl) solution (5g in 25 ml of water) each was again added to all the glass vials. All weighing with Mettler AC100 were gravimetrically controlled. Samples were gradually quenched by adding varying but increasing amount of the nitromethane as quenching agent then vigorously shaken and kept for stability. A blank sample was also prepared without the addition of KCl salt. All samples including blanks were carefully labelled and counted using Tricarb TR 1000 LSA each for 60 minutes to obtain good counting statistics. Records of count rates in counts per minutes (CPM) and Spectral index of the samples (SIS) as the quench indicating parameter (QIP) were taken. The total counting time for potassium chloride samples was more than 30 days in order to obtain stable counts rates.

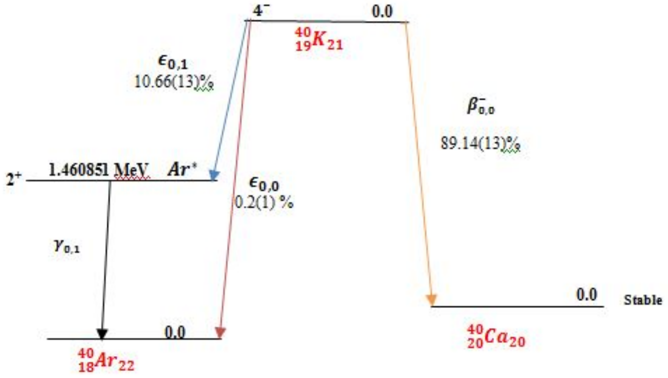


Figure 1: $^{40}_{19}K$ simplied decay scheme [4]

2.2. Liquid Scintillation Techniques

Radiation emitted from dissolved radionuclide samples in scintillation cocktails transferred energy to the organic scintillator that in turn emits light photons. This way each emission result is a pulse of light in form of digit called counts [13].

2.3. CIEMAT/NIST Model

The CIEMAT/NIST model used in this work for the calculation of ^{40}K specific activity was modified from Broda et al., [14] as shown in figure 2. The slight modification (improvement) is clearly reflected in figure 2 where four different polynomials were used to generate the empirical efficiencies of the tracer radionuclide (Tritium-3) using a method of interpolation called least squared fit analysis as stated below on Non-linear Least squared Fittings of Tritium calibration Curve. The usage of these polynomials for the fittings is to account for the least uncertainty associated with the empirically generated tracer efficiency as a result of the least squared fit analysis.

Broda et al description of CIEMAT/NIST method can be summarized into five steps as follows: (i) theoretical computation of detection efficiencies of both tracer and the radionuclide under study (^{40}K in this case) . This is done via computer program so as to establish an efficiency calibration curve (a plot of nuclide efficiency as function of tracer efficiency) [14]. (ii) Experimental determination of tracer efficiency from LSC measurement (CPM and QIPs). Since the activity (DPM) of the quench set is known, the efficiency of tracer is computed as (CPM/DPM). (iii) Measurement of samples of radionuclide under study for determination of tracer efficiencies and the corresponding radionuclide efficiencies. By applying the QIPs obtained from measured samples to equation (1) the tracer efficiencies are obtained. Similarly, by applying the obtained tracer efficiencies to equation (10), the corresponding radionuclide efficiencies are obtained. (iv) Conversion of the efficiency of the radionuclide so determined and the measured count rate (R$_n$) in CMP to the activity of the radionuclide [14].

R_n denotes sample count rate, μ is the free parameter, SIS is the spectral index of the sample as quench indicating parameter, m, is the mass of KCl salt in-cooperated into the 1 ml

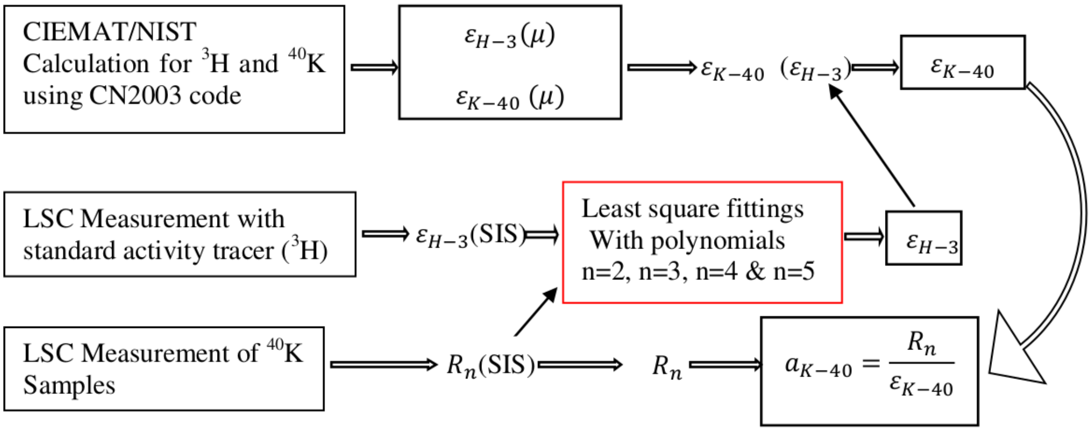


Figure 2: Illustration model for CIEMAT/NIST method [14].

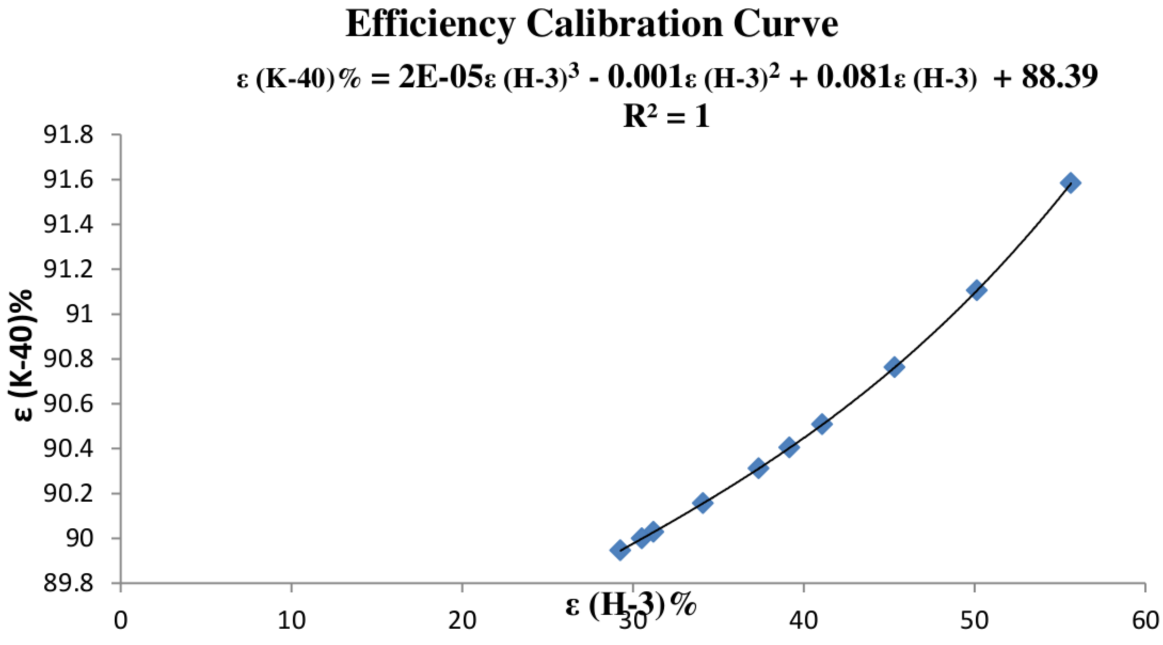


Figure 3: Efficiency calibration curve (a plot of efficiency of ^{40}K versus tracer efficiency)

of the sample $\varepsilon_{K-40}$and ε_{H-3} efficiencies of Potassium-40 and Tritium-3 radionuclides, respectively.

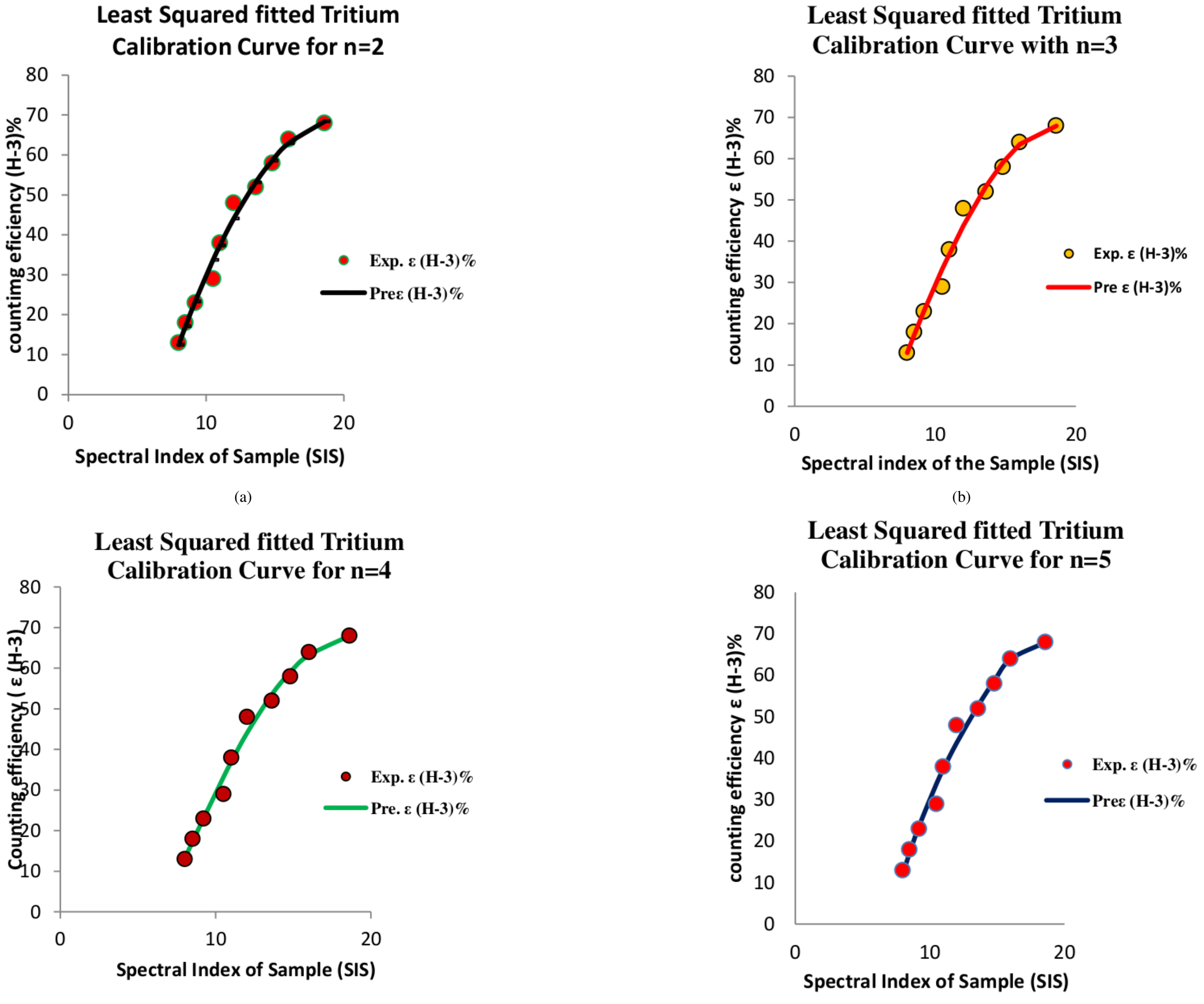
Normally, fittings of tracer calibration curves in CIMAT/NIST method are done using a single order polynomial [15]. However this study applied four (4) polynomials of different order to validate the tracer calibration curve hence generating four different sets of ^{40}K efficiencies. This served as the slight variation from other CIEMAT/NIST method.

2.4. *Analysis*

The significant analysis in this study is based on the nonlinear least squared fittings at different polynomial orders for tracer (Tritium) calibration curve as well as the evaluation of ^{40}K specific activity and half-life.

2.5. *Non-linear Least squared Fittings of Tritium calibration Curve*

Four least squared fitted calibration curves for tracer efficiency were obtained at varying polynomial order (n = 2, 3, 4, and 5) for tracer calibration curve (a plot of tracer efficiency ε_{H-3}versus quench indicating parameter). The tracer calibration curve upon which the current least square is fitted was obtained from radiation analysis [16]. The general form of polynomial used to generate the empirical tracer efficiencies for the least square fit analysis is given as equation (1) [17] while equations (2), (3), (4), and (10a) were used for the four different non-linear least square fittings in accordance with the polynomial order. All fittings were done using excel spread sheet including the calculations of relative standard uncertainties in the interpolated tracer efficiency associated with the least squared fittings using equations (7), (8) and (9) as presented in table 1



(a) (b) (c) (d)

Figure 4: Least squared fitted Tritium Calibration Curve for polynomials of order (a) $n = 2$; (b) $n = 3$; (c) $n = 4$ and (d); $n = 5$ respectively.

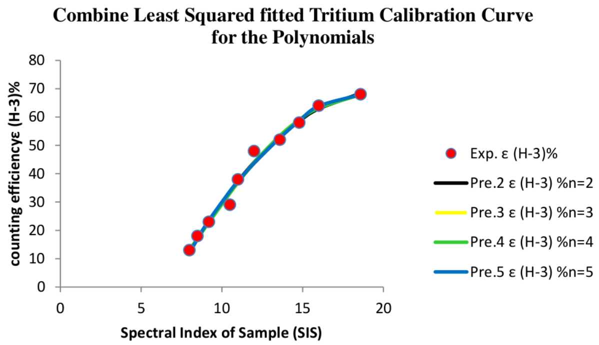


Figure 5: Combined least Squared Tritium calibration Curve for Polynomials ($n = 2$, $n = 3$, $n = 4$, $n = 5$)

and figures 3, 4, 5, and 6.

$$\varepsilon_{H-3} = \sum_{i=0}^{n} k_i (SIS)^i \tag{1}$$

For $n = 2$, the least squared theory becomes:

$$\varepsilon_{H-3} = k0 + k1\,(SIS) + k2(SIS)^2 \tag{2}$$

For $n = 3$ the least squared theory for fitting the tritium calibration curve becomes

$$\varepsilon_{H-3} = k_0 + k_1\,(SIS) + k_2(SIS)^2 + k_3(SIS)^3 \tag{3}$$

Similarly for $n = 4$ and $n = 5$ the theory for the least squared fitting of the calibration curve is given by

$$\text{å}_{H-3} = k_0 + k_1\,(SIS) + k_2(SIS)^2 + k_3(SIS)^3 + k_4(SIS)^4 \tag{4}$$

Table 1: Least Squared fit analysis, for Polynomials ($n = 2$, $n = 3$, $n = 4$, $n = 5$)

Experiment		Predicted	Least squared	Predicted	Predicted	Predicted	Least squared	Predicted	Least squared
		$n = 2$		$n = 3$		$n = 4$		$n = 5$	
SIS	Exp. ε(H-3)%	ε(H-3)%	Delta	ε(H-3)%	Delta squared	ε(H-3)%	Delta squared	ε(H-3)%	Delta squared
18.6	68	68.39	0.15217	67.939	0.00361	67.962	0.0014	67.949	0.00259
16	64	62.936	1.13039	63.507	0.24285	63.197	0.64414	63.865	0.01806
14.8	58	58.607	0.36942	59.058	1.12063	59.028	1.05738	58.71	0.50466
13.6	52	53.134	1.28618	53.286	1.65585	53.457	2.12396	52.707	0.50069
12	48	44.055	15.55986	43.775	17.84978	43.928	16.57536	43.708	18.41697
11	38	37.347	0.42523	36.909	1.18919	36.913	1.18076	37.343	0.43059
10.5	29	33.696	22.05335	33.242	17.9988	33.16	17.31165	33.863	23.65655
9.2	23	23.271	0.07371	23.066	0.0044	22.878	0.01482	23.585	0.34245
8.5	18	17.101	0.80676	17.249	0.56265	17.183	0.6673	17.151	0.71978
8	13	12.456	0.2955	12.971	0.00082	13.124	0.01553	12.108	0.79495
Sum			42.15261		40.628616		39.59230		45.38733
Standard Uncertainty(Su) in ε (H-3)%			0.684370		0.671884		0.663260		0.710143
Order of polynomial					Coefficients				
		k_0	k_1	k_2	k_3	k_4	k_5		
	$n = 2$	-88.89410	15.84820	-0.39742					
	$n = 3$	-61.11043	9.00344	0.14012	-0.013502				
	$n = 4$	-3.136618	-10.32740	2.48410825	-0.1360546	0.002333			
	$n = 5$	-104.8710	10.70044	2.43550	-0.387136	0.021350	-0.000419		

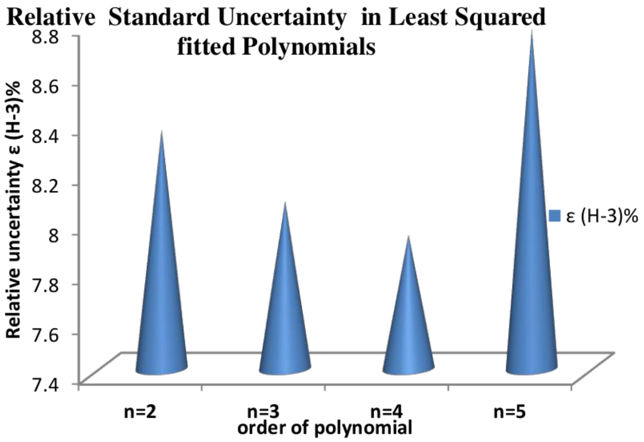


Figure 6: Relative uncertainty in tritium interpolated efficiencies for different polynomial

and

$$\varepsilon_{H-3} = k_0 + k_1\,(SIS) + k_2(SIS)^2 + k_3(SIS)^3 + k_4(SIS)^4 + k_5(SIS)^5, \tag{5}$$

where k_0, k_1, k_2, k_3, k_4, and k_5, are the coefficients of the least squared fittings respectively, SIS= Spectral index of the sample for tracer, and ε_{H-3} = tritium counting efficiency

$$Delta = Experiment - Theory \tag{6}$$

$$\text{Sum} = \text{Sum } of \text{ Delta Squared} \tag{7}$$

$$\text{Standard error} = SQRT\left(\frac{sum}{n*(n-1)}\right) \tag{8}$$

From the standard error, the relative standard uncertainties in empirically generated tritium efficiencies was obtained using equation (9) [18].

$$\frac{u(\varepsilon_{H-3})}{\varepsilon_{H-3}} = \sqrt{\sum\left(\frac{S\varepsilon}{\varepsilon_{H-3}}\right)^2} \tag{9}$$

2.6. Calculation of Efficiencies

All theoretical efficiencies for tracer (^{3}H) and ^{40}K were calculated using CN2003 code. The range of the calculation chosen for the tracer (Tritium) efficiency was between 25% to 65% for ionization quench constant of 0.0075 cm/MeV. The interpolated efficiency $(\varepsilon_{K-40})^{40}$K was obtained using a fitted polynomial given by equation (10) from the general form of equation given by [17] for some selected values of free parameter as shown in figure 3. The CN2003 visual basic code takes into account the following: statistics of nuclear decay, detector response as well as Physics of the nuclear decay processes.

$$\varepsilon_{K-40} = \sum_{i=0}^{n} k_i(\varepsilon_{H-3})^i \tag{10a}$$

$$\varepsilon(K-40)\% = 2E-05\varepsilon(H-3)^3 - 0.001\varepsilon(H-3)^2 + 0.081\varepsilon(H-3) + 88.39 \tag{10b}$$

The regression value of 1 on figure 3 indicates a perfect polynomial fitting.

2.7. ^{40}K specific Activity and Half-life

The measured SIS for Potassium samples were applied to the least squared fitted calibration curves to obtain the corresponding tracer (Tritium) efficiencies. The corresponding values of ^{40}K efficiencies (ε_{K-40}) were obtained by fitting the empirically generated tracer efficiencies to equation (10b). Equation (10b) is the efficiency calibration curve extracted from CIEMAT/NIST calculation for some selected values of free parameter (μ). With the efficiencies of ^{40}K (ε_{K-40}) obtained using equation (10b) and data of ^{40}K measurement from Liquid Scintillation counter (Count rates and SIS), the specific activities of ^{40}K in all the measured samples were computed using equation (11) as follows [4]:

$$a = \frac{R_s - R_b}{m\varepsilon_{K-40}}, \tag{11}$$

Table 2: Specific activities, half-lives, decay constants and efficiencies for all KCl samples

Order of Polynomial	Sample ID	Efficiency		Specific Activity	Half-life ($T_{1/2}$)	Decay constant (λ)
		$\epsilon(H-3)\%$	$\epsilon(K-40)\%$	Bq/g	$\times 10^9\ y$	$\times 10^{-10} y^{-1}$
$n = 2$	GVT1	60.6755	94.0907	12.916	1.6031	4.3234
	GVT2	49.764	92.4092	16.247	1.2745	5.4344
	GVT3	46.6121	92.0183	17.011	1.2173	5.6908
	GVT4	46.7852	92.0388	17.651	1.1731	5.9048
	GVT5	50.6193	92.5219	15.839	1.3073	5.2981
	GVT6	46.2459	91.9753	18.741	1.1049	6.2697
	GVT7	49.2336	92.3407	16.556	1.2507	5.5381
	GVT8	47.2002	92.0884	16.637	1.2447	5.5555
	GVT9	48.5306	92.2517	17.376	1.1917	5.8125
			Mean	16.541	1.2518	5.5365
$n = 3$	GVT1	61.2099	94.1879	12.91	1.6047	4.3189
	GVT2	49.7335	92.4052	16.236	1.2754	5.4346
	GVT3	46.432	91.9971	17.005	1.2177	5.692
	GVT4	46.6126	92.0184	17.645	1.1736	5.9061
	GVT5	50.6334	92.5237	15.828	1.3083	5.298
	GVT6	46.0501	91.9525	18.736	1.1052	6.271
	GVT7	49.1763	92.3334	16.546	1.2514	5.5385
	GVT8	47.046	92.0699	16.63	1.2452	5.5562
	GVT9	48.4386	92.2402	17.367	1.1923	5.8132
			Mean	16.540	1.2519	5.5364
$n = 4$	GVT1	61.0546	94.1595	12.907	1.6043	4.3204
	GVT2	49.2672	92.345	16.232	1.2757	5.4331
	GVT3	46.168	91.9662	17.002	1.2179	5.6908
	GVT4	46.336	91.9858	17.651	1.1738	5.9047
	GVT5	50.1258	92.4565	15.834	1.3086	5.2965
	GVT6	45.8133	91.9251	18.731	1.1055	6.2699
	GVT7	48.7391	92.2779	16.542	1.2518	5.537
	GVT8	46.7397	92.1912	16.545	1.2478	5.5547
	GVT9	48.04352	92.1912	17.363	1.1926	5.8117
			Mean	16.537	1.2521	5.5354
$n = 5$	GVT1	61.1382	94.1748	12.905	1.6045	4.3195
	GVT2	49.2672	92.34508	16.247	1.2745	5.4388
	GVT3	46.168	91.96626	17.011	1.2173	5.6941
	GVT4	46.336	91.98589	17.651	1.1731	5.9082
	GVT5	50.1258	92.45652	15.839	1.3073	5.3019
	GVT6	45.8133	91.92513	18.741	1.1049	6.2731
	GVT7	48.7391	92.27796	16.556	1.2507	5.5418
	GVT8	46.7397	92.03347	16.637	1.2447	5.5592
	GVT9	48.0435	92.19121	17.376	1.1917	5.5816
			Mean	16.548	1.2513	5.5391

where R_s =Sample count rate, R_b = Background count rate, m = mass of potassium salt incorporated into the sample, ε_{K-40} = counting efficiency for ^{40} K.

From the specific activity obtained using equation (8) the half-life of ^{40} K was computed using equation (12) [4]:

$$T_{1/2} = \frac{(\ln 2)\frac{N_A}{M}p}{a}, \tag{12}$$

where N_A = Avogadro constant, M is the relative molar mass of KCl, $p = N(^{40}_{19}K)/N(K) = 0.01167\%$.

Following the relationship between half-life and decay constant, the decay constant was computed using equation (13) expressed as [11]:

$$\lambda = \frac{\ln 2}{T_{1/2}} \tag{13}$$

3. Results and Discussion

Results of Least Squared fitted Tritium Calibration Curves for all the polynomials is presented in figures 4 and 5.

From figure 4 (a, b, c and d), the least squared fitted curves of the experimental tritium calibration curve it can be observed

Table 3: ^{40}KHalf-life and decay constant values from source and some calculated decay constants

Reference	^{40}K ($T_{1/2}$) $\times 10^9 y$	^{40}K (λ) $\times 10^{-10} y^{-1}$	
	Original	Original	Calculated
Steiger and Jägar, (1977) [20]	1.250	5.543	-
Min et al. (2000) [19]	1.269	5.463	-
Grau Malonda and Grau Carles (2002) [21]	1.248	-	5.554
Kossert and Günther,(2004) [4]	1.248	-	5.554
This study (polynomial order)			
$n = 2$	1.2519	5.5367	5.5367
$n = 3$	1.2518	5.5365	5.5365
$n = 4$	1.2521	5.5354	5.5354
$n = 5$	1.2513	5.5391	5.5391

that not all points of empirically generated efficiencies are perfectly fitted to the experimental data. This is because each fitted polynomial has an associated level of uncertainty in determining the empirical efficiencies for interpolation with the spectral index of Potassium Chloride (KCL) measured samples as clearly shown in figure 5.

The magnitude of the standard uncertainties associated with each polynomial including the relative standard interpolated efficiencies for the least squared analysis is shown is Table 1.

All least squared fit analysis in this study and the estimation of standard and relative interpolated efficiencies of Tritium were carried out using Excel spread sheet. From table 1 it can be seen that the standard uncertainties and relative standard uncertainties in tritium interpolated efficiencies due to the Least squared fitted polynomials ranges from (0.663260 to 0.710143)% and (7.941 to 8.767)% respectively. Polynomial of order ($n = 4$) has the lowest interpolation uncertainties while the highest uncertainty was associated to polynomial of n=5 as clearly shown in figure 6. It can also be deduced from figure 5 that the uncertainties associated with non-linear least square fit analysis of the interpolated efficiencies does not completely depend on the polynomial order.

3.1. ^{40}K Specific Activity, Half-life and Decay Constant

The values of ^{40}K specific activity, as well as the computed half-life and decay constants obtained in this study for the four (4) different polynomials are presented in table 2.

From results presented in table 2, it can be seen that the mean obtained values of ^{40}K specific activity, Half-life and decay constant for the four least squared fitted polynomials varied slightly ranging from (16.537 to 16.548) Bq/g, (1.2513 to1.2521) $\times 10^9$ y and (5.5354 to 5.5392)$\times 10^{-10} y^{-1}$ respectively. This slight variation can be linked to the relative standard uncertainties associated with empirically generated interpolation efficiencies of the tracer (Tritium) from the least squared fitted polynomials. Polynomial of n=4 has the lowest specific activity value of 16.537 Bq/g followed by Polynomial of $n = 3$ with 16.540 Bq/g, then polynomial of $n = 2$ (which is basically quadratic) with 16.541 Bq/g and lastly polynomial of $n = 5$ with the highest value of16.548.This results shows that the higher the relative uncertainty associated with the least squared polynomial fitting, the higher the values of specific activities and decay constants. On the contrary, higher values of half-life are linked to lower relative standard interpolated uncertainties of tracer (Tritium) associated with the least squared fitted polynomials. However, all values of specific activities in this current study are in good agreement with values of (16,594 to 16.616) Bq/g obtained by Kossert and Günther, [4] while the half-life and decay constant values are in good agreement with the values obtained by Steiger and Jäger, Min *et al.*, Grau Malonda and Grau Carles, Kossert and Güther, [4, 19, 20, 21] as shown in table 3.

4. Conclusion

The relative standard uncertainties associated with the interpolated tracer efficiencies from the least squared fitting of the four different polynomials used in this study revealed that the choice of the prefer polynomial fit does not necessary depend on the order of polynomial but on the coefficients of the polynomial that will produce the lowest standard error. This is obtained through control trials starting from the lowest order. The validity of this is seen in the slight variation of ^{40}K specific activity, upon which half-life is computed as well as decay constant. The slight variation in these values is as a result of the closed agreement in the relative standard uncertainties of the fitted polynomials. However a more noticeable variation is observed with least squared fitted polynomial of n=5. The choice of the best fit will go a long way in further reducing the relative uncertainty associated with interpolated tracer efficiency, which could improve the accuracy of CIEMAT/NIST method in the activity determination of radionuclide and strengthen its acceptance. Also, further non-linear least square fit analysis of tracer (Tritium) calibration curve should be carried out with polynomials of higher order to confirm the independence of the best fit on the order of polynomial.

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