

SPREADSHEET DESIGN AND VALIDATION FOR CHARACTERISTIC LIMITS DETERMINATION IN GROSS ALPHA AND BETA MEASUREMENT

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ABSTRACT

The identification and detection of ionizing radiation are essential requisites of radiation protection. Gross alpha and beta measurements are widely applied as a screening method in radiological characterization, environmental monitoring and industrial applications. As in any other analytical technique, test performance depends on the quality of instrumental measurements and reliability of calculations. Characteristic limits refer to three specific statistics, namely, decision threshold, detection limit and confidence interval, which are fundamental to ensuring the quality of determinations. This work describes a way to calculate characteristic limits for measurements of gross alpha and beta activity applying spreadsheets. The approach used for determination of decision threshold, detection limit and limits of the confidence interval, the mathematical expressions of measurands and uncertainty followed standards guidelines. A succinct overview of this approach and examples are presented and spreadsheets were validated using specific software. Furthermore, these spreadsheets could be used as tool to instruct beginner users of methods for ionizing radiation measurements.

1. INTRODUCTION

Human exposure to radiation has been a subject of great interest for public health. For instance, guidelines from the World Health Organization [1] establish that gross alpha and beta activity should be monitored in waters for human consumption. The recommended levels are 0.5 Bq L^{-1} for gross alpha and 1 Bq L^{-1} for gross beta activities. Such activities are related to Individual Dose Criterion (IDC) of 0.1 mSv from 1 year's consumption of drinking-water that would usually not be exceeded if gross alpha and beta activity were equal to or below these recommended levels.

Gross alpha and gross beta analyses are often used to screen samples to determine if further specific radionuclide analyses are required. Indeed, these analyses are used in radiological environmental monitoring as the first step for characterizing radioactivity in drinking water. However, a wide range of other matrices, such as wastewaters, air filters and soils can be evaluated. In general, simple radioanalytical procedures are used to measure gross alpha and gross beta. For example, the sample source can be prepared by co-precipitation or evaporation methods and then the radioactivity measurement of the residue deposited on a planchet is performed using proportional detector or liquid scintillation counting.

There are several procedures and ISO standards for gross alpha and beta determinations in water matrices [2-6]. However, DOE RP710 [7] describes a more comprehensive method

regarding the type of matrix. That method is used to rapidly screen high and low activities of alpha and beta emitting radionuclides in waters, air filters, soils, sludges, wastewaters, and solvents. The sample can be leached with acids and, if necessary, organic matter oxidized. An aliquot of the sample or leachate is then evaporated to dryness on a stainless steel planchet and counted for alpha and/or beta radioactivity on a low-background gas-flow proportional counter. Alpha and beta activities depend on sample mass residue in the steel planchet and are based on calibration of specific isotopic standards, such as Am-241 and Sr-90/Y-90 certified sources. Interferences and method limitations, apparatus, materials, reagents, sample collection and preservation, matrix specific procedure and calculations including crosstalk parameters are also discussed in that document. The results are alpha and beta activity and their respective uncertainties. The calculation of characteristic limits confidence interval, decision threshold and detection limit, which are fundamental parameters to ensuring the quality of determinations, are not indicated. Nevertheless, the calculation included in other procedures and standards are supposed to be used. The APHA method 7110 [2] reports the radioactivity concentration using a confidence level of 95% to express the confidence interval and Currie method to estimate the limit of detection. The ISO standards for gross alpha and beta determination refer to ISO 11929 [8] to calculate the characteristic limits.

In general, activity, uncertainty, confidence interval and limit of detection parameters of gross alpha and beta determinations may be provided by software of counter instrument used in the measurement [9,10]. If this is not available, these parameters may be obtained using spreadsheets designed by the user. For that reason, the aim of this work is offer an approach to assist users in the task of spreadsheets construction for measurand, uncertainty and characteristic limits calculations, used in gross alpha and beta determinations, using standard guidelines.

2. MATERIAL AND METHODS

Measurement functions of gross alpha and beta determinations were obtained in DOE RP710 [7]. Determination of characteristic limits decision threshold, detection limit and limits of the confidence interval for measurements of gross alpha and beta were performed using ISO 11929 [8]. The spreadsheet has been developed for Microsoft® Excel Office 2007 [11], and is compatible with BrOffice.Org Open Office® [12]. The use of spreadsheet is demonstrated with an example of water sample analysis. Spreadsheets validation were made with UncertRadio (Version 1.07 2013/03) software [13].

3. RESULTS AND DISCUSSIONS

In order to perform gross alpha and beta determinations following DOE RP710, an aliquot size of sample is transferred quantitatively to a counting planchet, dried, the net residue mass is determined and counted for an appropriate time in a detector system.

Some instrumental parameters are necessary to ensure accurate measurements, namely, detector efficiency and crosstalk as a function of sample mass. They are equivalent to determining a zero-mass efficiency with an associated self absorption factor.

Due to the didactical objective of this work, all parameters will refer to a same specific residue mass of standard and sample. On the contrary, four calibration curves (for alpha efficiency, beta efficiency, alpha to beta crosstalk, and beta to alpha crosstalk) as a function of residue mass of standard must to be obtained so that the data for any arbitrary sample residue mass may be interpolated from these curves [7].

Using data from calibration standards with a specific residue mass (m), the efficiencies are calculated as follows:

$$\varepsilon_{\alpha(m)} = \frac{N_{\alpha_ast(m)}}{D_{\alpha st}} \quad (1)$$

$$\varepsilon_{\beta(m)} = \frac{N_{\beta_bst(m)}}{D_{\beta st}} \quad (2)$$

Where:

$\varepsilon_{\alpha(m)}$ = measured alpha efficiency for residue mass (m)

$N_{\alpha_ast(m)}$ = measured alpha net count rate of alpha standard for residue mass (m)

$D_{\alpha st}$ = known disintegration rate of alpha standard

$\varepsilon_{\beta(m)}$ = measured beta efficiency for residue mass (m)

$N_{\beta_bst(m)}$ = measured beta net count rate of beta standard for residue mass (m)

$D_{\beta st}$ = known disintegration rate of beta standard

The same data used to generate the efficiencies can also be used to generate the crosstalk factors, as follows:

$$X_{\beta\alpha(m)} = \frac{N_{\alpha_bst(m)}}{N_{\beta_bst(m)}} \quad (3)$$

$$X_{\alpha\beta(m)} = \frac{N_{\beta_ast(m)}}{N_{\alpha_ast(m)}} \quad (4)$$

Where:

$X_{\beta\alpha(m)}$ = beta to alpha crosstalk for residue mass (m)

$N_{\beta_ast(m)}$ = measured beta net count rate of alpha standard for residue mass (m)

$X_{\alpha\beta(m)}$ = alpha to beta crosstalk for residue mass (m)

$N_{\alpha_bst(m)}$ = measured alpha net count rate of beta standard for residue mass (m)

Sample count data, efficiencies and crosstalk factors are used to calculate the alpha and beta activity using the following equations:

$$N_{\alpha(m)} = \frac{C_{\alpha}}{t_{C_{\alpha}}} - \frac{B_{\alpha}}{t_{B_{\alpha}}} \quad (5)$$

$$N_{\beta(m)} = \frac{C_{\beta}}{t_{C_{\beta}}} - \frac{B_{\beta}}{t_{B_{\beta}}} \quad (6)$$

$$N_{X\alpha(m)} = N_{\alpha(m)} - X_{\beta\alpha(m)}N_{\beta} = \left(\frac{C_{\alpha}}{t_{C_{\alpha}}} - \frac{B_{\alpha}}{t_{B_{\alpha}}} \right) - X_{\beta\alpha(m)} \left(\frac{C_{\beta}}{t_{C_{\beta}}} - \frac{B_{\beta}}{t_{B_{\beta}}} \right) \quad (7)$$

$$\varepsilon_{X\alpha(m)} = \varepsilon_{\alpha(m)}(1 - X_{\alpha\beta(m)}X_{\beta\alpha(m)}) \quad (8)$$

$$A_{\alpha} = \frac{N_{X\alpha(m)}}{\varepsilon_{X\alpha(m)}Q} = \frac{\left(\frac{C_{\alpha}}{t_{C_{\alpha}}} - \frac{B_{\alpha}}{t_{B_{\alpha}}} \right) - X_{\beta\alpha(m)} \left(\frac{C_{\beta}}{t_{C_{\beta}}} - \frac{B_{\beta}}{t_{B_{\beta}}} \right)}{\varepsilon_{\alpha(m)}(1 - X_{\alpha\beta(m)}X_{\beta\alpha(m)})Q} \quad (9)$$

$$N_{X\beta(m)} = N_{\beta(m)} - X_{\alpha\beta(m)}N_{\alpha} = \left(\frac{C_{\beta}}{t_{C_{\beta}}} - \frac{B_{\beta}}{t_{B_{\beta}}} \right) - X_{\alpha\beta(m)} \left(\frac{C_{\alpha}}{t_{C_{\alpha}}} - \frac{B_{\alpha}}{t_{B_{\alpha}}} \right) \quad (10)$$

$$\varepsilon_{X\beta(m)} = \varepsilon_{\beta(m)}(1 - X_{\alpha\beta(m)}X_{\beta\alpha(m)}) \quad (11)$$

$$A_{\beta} = \frac{N_{X\beta(m)}}{\varepsilon_{X\beta(m)}Q} = \frac{\left(\frac{C_{\beta}}{t_{C_{\beta}}} - \frac{B_{\beta}}{t_{B_{\beta}}} \right) - X_{\alpha\beta(m)} \left(\frac{C_{\alpha}}{t_{C_{\alpha}}} - \frac{B_{\alpha}}{t_{B_{\alpha}}} \right)}{\varepsilon_{\beta(m)}(1 - X_{\alpha\beta(m)}X_{\beta\alpha(m)})Q} \quad (12)$$

Where:

$N_{\alpha(m)}$ = measured alpha net count rate of sample for residue mass (m)

C_{α} = measured alpha sample counts

$t_{C_{\alpha}}$ = alpha sample count time

B_{α} = measured alpha background counts

$t_{B_{\alpha}}$ = alpha background count time

$N_{\beta(m)}$ = measured beta net count rate of sample for residue mass (m)

C_{β} = measured beta sample counts

$t_{C_{\beta}}$ = beta sample count time

B_{β} = measured beta background counts

$t_{B_{\beta}}$ = beta background count time

$N_{X\alpha(m)}$ = crosstalk corrected net alpha count rate of sample for residue mass (m)

$\varepsilon_{X\alpha(m)}$ = crosstalk corrected alpha efficiency at residue mass (m)

A_{α} = alpha activity

Q = sample size (volume or mass)

$N_{X\beta(m)}$ = crosstalk corrected net beta count rate of sample for residue mass (m)

$\varepsilon_{X\beta(m)}$ = crosstalk corrected beta efficiency at residue mass (m)

A_{β} = beta activity

Beyond sample activity concentration, the uncertainty associated with its measurement process as well as its characteristics limits, which are the statistical limits that determine if the result is adequate to determine the existence of radioactivity in the measured sample, must be evaluated. The treatment of measurement uncertainties was carried out by means of the general procedures according to the ISO Guide to the Expression of Uncertainty in Measurement [14] and the successive determination of the characteristic limits by using the standard uncertainty obtained from the evaluation, according to ISO 11929 [8].

In the gross alpha determination example, the measurand alpha activity (Y) is a function of several input quantities (X_i ; where $i = 1, \dots, m$):

$$Y = G(X_1 \dots X_m) = (X_1 - X_2 X_3 - X_4) \frac{X_6 \dots}{X_5 X_7 \dots} = (X_1 - X_2 X_3 - X_4) W \quad (13)$$

$$W = \frac{X_6 \dots}{X_5 X_7 \dots} \quad (14)$$

G is the model function, m is the number of input quantities, X_1 is the gross count rate and X_2 is the background count rate. The other input quantities X_i are calibration, correction factors or influence quantities. The x_i are estimates of the input quantities X_i . If some of these input quantities are not involved, such as X_3 in this example, the estimate is $x_i = 1$ (so $x_3 = 1$) and its uncertainty is $u(x_i) = 0$ (so $u(x_3) = 0$).

In this example, the estimates x_i are: $x_1 = C_\alpha/t_{C\alpha}$, $x_2 = B_\alpha/t_{B\alpha}$, $x_3 = 1$, $x_4 = X_{\beta\alpha(m)}(C_\beta/t_{C\beta} - B_\beta/t_{B\beta})$, $x_5 = \epsilon_{X\beta(m)}$, $x_6 = 1$, and $x_7 = Q$. Substituting the input quantity by estimates x_i in equations above, the primary estimate y of the measurand Y results:

$$y = G(x_1 \dots x_m) = (x_1 - x_2 x_3 - x_4) w = \left[\frac{C_\alpha}{t_{C\alpha}} - \frac{B_\alpha}{t_{B\alpha}} - X_{\beta\alpha(m)} \left(\frac{C_\beta}{t_{C\beta}} - \frac{B_\beta}{t_{B\beta}} \right) \right] \frac{1}{\epsilon_{X\alpha(m)} Q} \quad (15)$$

The standard uncertainty $u(y)$ of the measurand associated with the primary measurement result y follows from the relation:

$$u^2(y) = \sum_{i=1}^m \left(\frac{\partial G}{\partial X_i} \right)^2 u^2(x_i) \quad (16)$$

Using the respective partial derivatives

$$\frac{\partial G}{\partial X_1} = W; \quad \frac{\partial G}{\partial X_2} = -X_3 W; \quad \frac{\partial G}{\partial X_3} = -X_2 W; \quad \frac{\partial G}{\partial X_4} = -W; \quad \frac{\partial G}{\partial X_i} = \pm \frac{Y}{X_i} \quad (i \geq 5) \quad (17)$$

and by substituting the estimates x_i , w and y, the standard uncertainty $u(y)$ of the measurand associated with y is :

$$u(y) = \sqrt{w^2[u^2(x_1) + x_3^2 u^2(x_2) + x_2^2 u^2(x_3) + u^2(x_4)] + y^2 u_{\text{rel}}^2(w)} \quad (18)$$

Where the squared standard uncertainties of estimates x_i are:

$$u^2(x_1) = \frac{C_\alpha}{t_{C_\alpha}^2} \quad (19)$$

$$u^2(x_2) = \frac{B_\alpha}{t_{B_\alpha}^2} \quad (20)$$

$$u^2(x_3) = 0 \quad (21)$$

$$u^2(x_4) = X_{\beta\alpha(m)}^2 \left(\frac{C_\beta}{t_{C_\beta}^2} + \frac{B_\beta}{t_{B_\beta}^2} \right) + u^2_{X_{\beta\alpha(m)}} \left(\frac{C_\beta}{t_{C_\beta}} - \frac{B_\beta}{t_{B_\beta}} \right)^2 \quad (22)$$

$$u_{\text{rel}}^2(w) = \sum_{i=5}^m \frac{u^2(x_i)}{x_i^2} = \left(\frac{u_{\varepsilon_{X\alpha(m)}}}{\varepsilon_{X\alpha(m)}} \right)^2 + \left(\frac{u_Q}{Q} \right)^2 \quad (23)$$

$$u_{\varepsilon_{X\alpha(m)}}^2 = \left(1 - X_{\alpha\beta(m)} X_{\beta\alpha(m)} \right)^2 u_{\varepsilon_{\alpha(m)}}^2 + \varepsilon_{\alpha(m)}^2 X_{\beta\alpha(m)}^2 u_{X_{\alpha\beta(m)}}^2 + \varepsilon_{\alpha(m)}^2 X_{\alpha\beta(m)}^2 u_{X_{\beta\alpha(m)}}^2 \quad (24)$$

As defined in ISO 11929:

Decision threshold (y^*) is the “value of the estimator of the measurand, which when exceeded by the result of an actual measurement using a given measurement procedure of a measurand quantifying a physical effect, one decides that the physical effect is present”.

Detection limit ($y^\#$) is the “smallest true value of the measurand which ensures a specified probability of being detectable by the measurement procedure”.

For the calculation of these limits, the standard uncertainty of the measurand is needed as a function $\tilde{u}(\tilde{y})$ of its true value, \tilde{y} , as follows:

$$\tilde{u}^2(\tilde{y}) = c_0 + c_1 \tilde{y} + c_2 \tilde{y}^2 \quad (25)$$

c_0 , c_1 and c_2 are the coefficients of a second-order polynomial. From equation (18), the following values are:

$$c_0 = w^2 \left[\left(\frac{B_\alpha}{t_{B_\alpha}} \frac{x_3}{t_{C_\alpha}} \right) + \left(\frac{B_\alpha}{t_{B_\alpha}} \frac{x_3^2}{t_{B_\alpha}} \right) + \left(\frac{B_\alpha}{t_{B_\alpha}} u^2(x_3) \right) + \frac{x_4}{t_{C_\alpha}} + u^2(x_4) \right] \quad (26)$$

$$c_1 = \frac{w}{t_{C_\alpha}} \quad (27)$$

$$c_2 = u_{\text{rel}}^2(w) \quad (28)$$

If the probability α of the error of the first kind and probability β of the error of the second kind are chosen to be equally, the quantiles of the standard normal distribution for the probability $1 - \alpha$ and $1 - \beta$, may be written as $k_{1-\alpha} = k_{1-\beta} = k$, and the limits are calculated as:

$$y^* = k\sqrt{c_0} \quad (29)$$

$$y^\# = \frac{2y^* + k^2 c_1}{1 - k^2 c_2} \quad (30)$$

If the primary measurement result (y) exceeds the decision threshold (y^*), the physical effect is recognized as present. In such way the limits of the confidence interval contains the true value of the measurand with the specified probability $1 - \gamma$. The lower limit (y^\blacktriangleleft) and upper limit (y^\blacktriangleright) of the confidence interval are provided by:

$$y^\blacktriangleleft = y - k_p u(y) \quad \text{with } p = \omega \left(1 - \frac{\gamma}{2} \right) \quad (31)$$

$$y^\blacktriangleright = y - k_q u(y) \quad \text{with } q = 1 - \omega \frac{\gamma}{2} \quad (32)$$

Where ω is standardized normal distribution of the measurand to its standard uncertainty ratio:

$$\omega = \frac{1}{\sqrt{2\pi}} \int_{-\infty}^{\frac{y}{u(y)}} \exp\left(-\frac{v^2}{2}\right) dv = \Phi\left[\frac{y}{u(y)}\right] \quad (33)$$

In ISO 11929, it is considered that non-negative measurand shall be assigned to the physical effect and this is taken into account by the best estimate of the measurand (\hat{y}) given by:

$$\hat{y} = y + \frac{u(y) \exp\left(\frac{-y^2}{2u^2(y)}\right)}{\omega\sqrt{2\pi}} \quad (34)$$

The standard uncertainty associated with \hat{y} is given by:

$$u(\hat{y}) = \sqrt{u^2(y) - \hat{y}(\hat{y} - y)} \quad (35)$$

The same approach was applied for gross beta determination example. The equations describe above were used to build four spreadsheets and data for water sample analysis were used to illustrate their application, as presented below. The spreadsheets are self-explanatory and just the blue cells need be filled out with standard, sample and probability data. Fig. 1 shows the spreadsheet where the alpha and beta standard and sample input data are set.

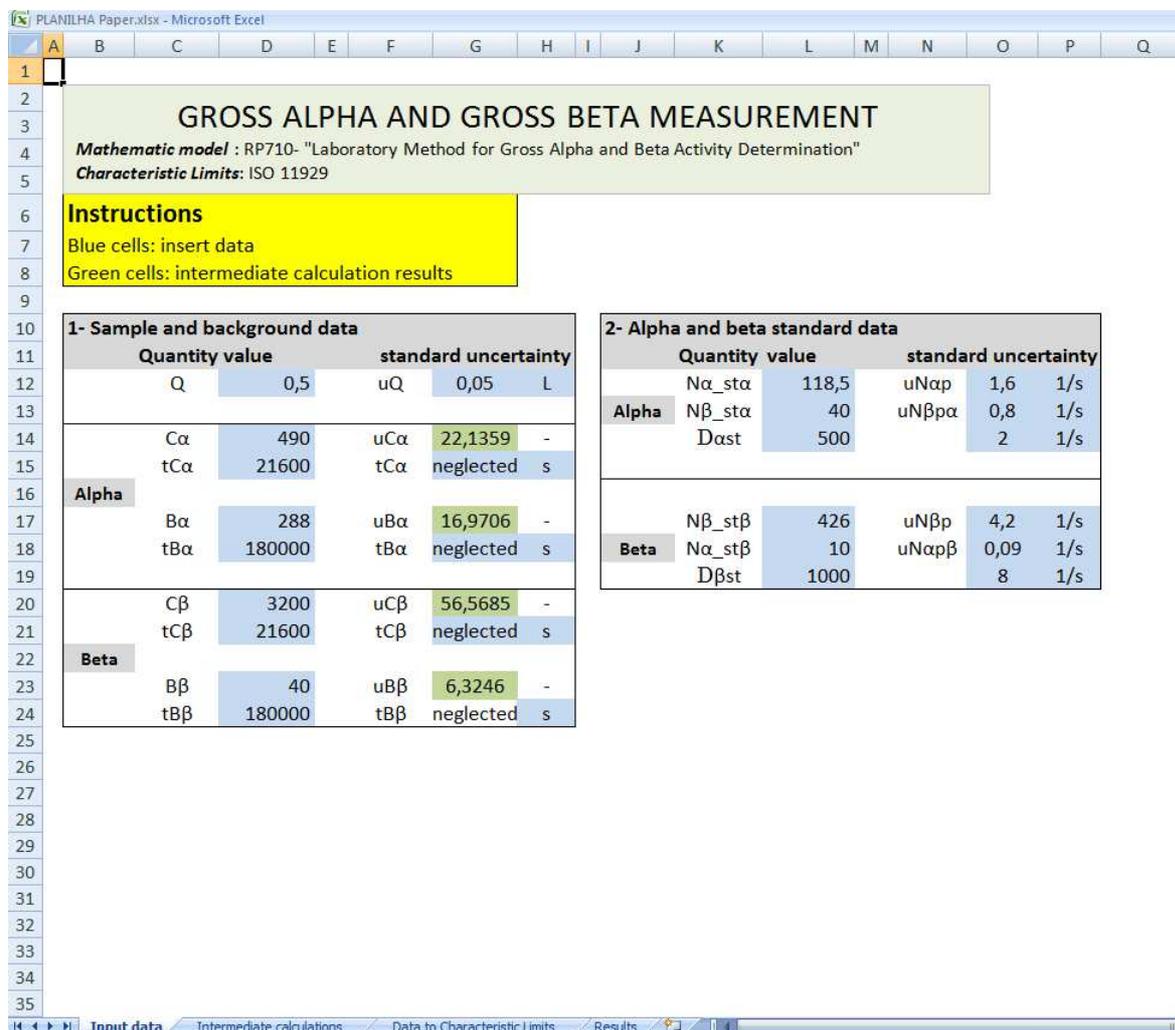


Figure 1 – Input data spreadsheet.

Fig. 2 shows the spreadsheet used for intermediate calculations, such as gross and background rates, crosstalk correction factors, efficiencies and crosstalk corrected efficiencies.

PLANILHA Paper.xlsx - Microsoft Excel

GROSS ALPHA AND GROSS BETA MEASUREMENT
Mathematic model : RP710- "Laboratory Method for Gross Alpha and Beta Activity Determination"
Characteristic Limits: ISO 11929

Instructions
Green cells: intermediate values

3- Intermediate values

Quantity	value	standard uncertainty		
C α /tC α	0,02269	uC α /tC α	0,001024812	1/s
B α /tB α	0,0016	uB α /tB α	9,42809E-05	1/s
C β /tC β	0,14815	uC β /tC β	0,002618914	1/s
B β /tB β	0,00022	uB β /tB β	3,51364E-05	1/s
X $\beta\alpha$ (m)	0,02347	uX $\beta\alpha$ (m)	0,000313363	
X $\alpha\beta$ (m)	0,33755	uX $\alpha\beta$ (m)	0,008145498	
$\epsilon\alpha$ (m)	0,237	u $\epsilon\alpha$ (m)	0,0033375	
$\epsilon\beta$ (m)	0,426	u $\epsilon\beta$ (m)	0,0054087	
$\epsilon\alpha\alpha$ (m)	0,23512	u $\epsilon\alpha\alpha$ (m)	0,003311429	
$\epsilon\alpha\beta$ (m)	0,42262	u $\epsilon\alpha\beta$ (m)	0,005366689	

Input data Intermediate calculations Data to Characteristic Limits Results

Figure 2 – Intermediate calculations spreadsheet.

Fig. 3 shows the spreadsheet where data to characteristic limits are set and calculated, such as input data x_1 to x_7 and respective standard uncertainties, sensitivity factor (w) and its squared relative uncertainty ($u_{rel}^2(w)$), and parameters c_0 , c_1 and c_2 , used in decision threshold detection limit calculation.

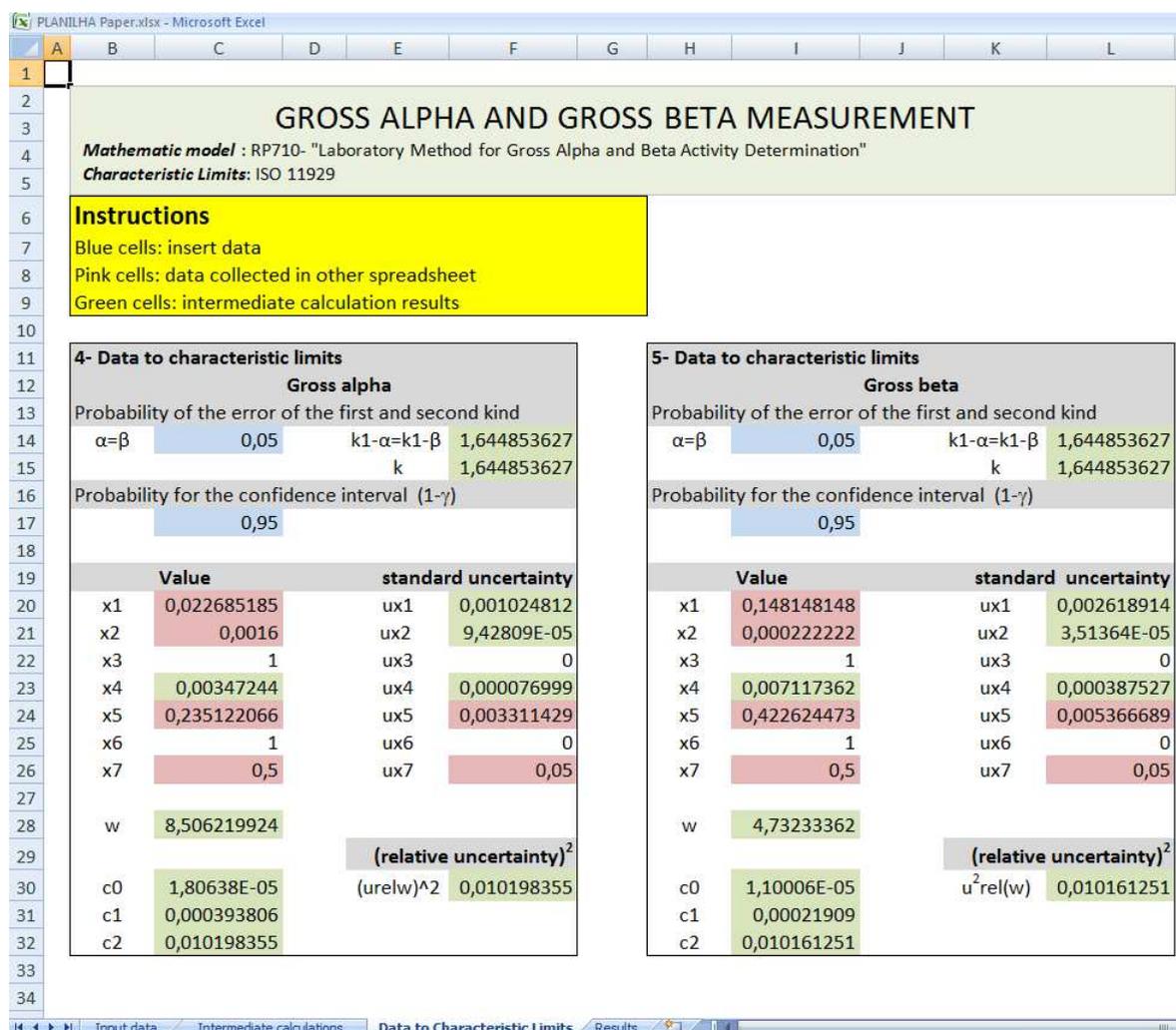


Figure 3 – Data to characteristic limits spreadsheet.

Fig. 4 shows the spreadsheet where results for primary measurement result (y) and the respective standard uncertainty associated ($u(y)$), decision threshold (y^*), detection limit ($y^\#$), lower limit (y^\blacktriangleleft) and upper limit of the confidence interval (y^\blacktriangleright), best estimate of the measurand y (\hat{y}) and the respective standard uncertainty associated ($u(\hat{y})$) are calculated.

Calculations used in these spreadsheets were validated by using of UncertRadio (Version 1.07 2013/03) software [13], that allows the automated calculation of the value of the output quantity, its assigned combined measurement uncertainty (according to ISO GUM), and characteristic limits according to ISO 11929. All calculated value agreed and the validation results are showed in purple cells beside each calculated values of measurand, standard uncertainty and characteristic limits.

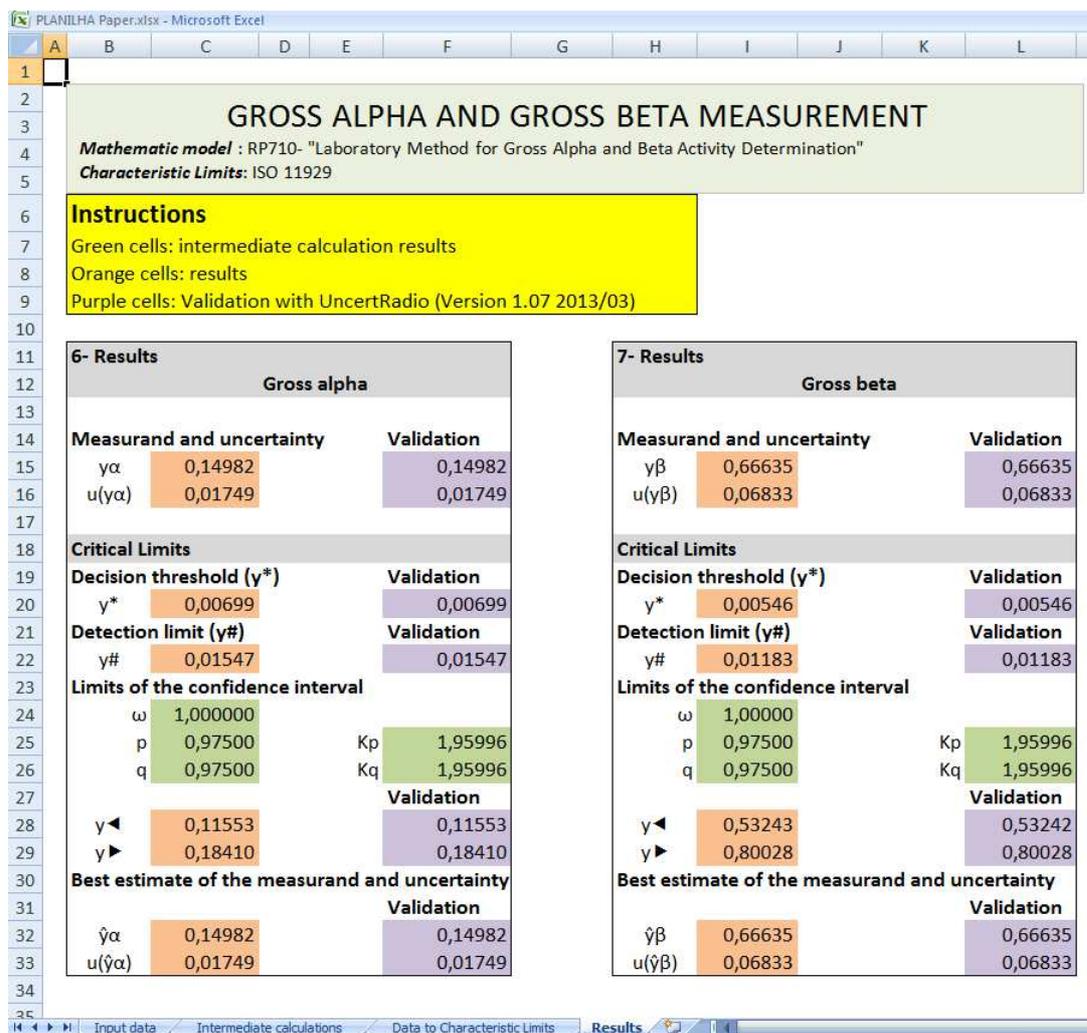


Figure 4 – Results spreadsheet.

4. CONCLUSIONS

In this work, we demonstrated an approach to construct spreadsheets for calculation of measurand and the respective standard uncertainty and characteristic limits of gross alpha and gross beta determination. The mathematical model was based in DOE RP710 and characteristic limits calculations follow ISO 11929 standard, that is with GUM recommendations. For high sample throughput in any analytical determination, using closed software calculations presents advantages. On the other hand, inexperienced users may take more benefit using spreadsheets as tool, at first time, when they are in instruction stage, as they would learn about the calculation process. For this reason, we expect the approach described here will be useful for beginner users of gross alpha and beta methods determinations.

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