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Detection limit calculation according to ISO 11929 for in vitro ^{210}Pb radiobioassay determinations by LSC

Sampaio[a], C.S.; Martins[a], M.M.; Sousa[a], W.O.

[a] *DIDOS/IRD/CNEN, 22783-127, Rio de Janeiro, RJ, Brazil*

camilla.sampaio@ird.gov.br

ABSTRACT

According to the limit of detection (DL) presented in ISO 11929:2010, the International Organization for Standardization (ISO) abandoned the concept of minimum detectable amount (MDA) described in ISO 12790-1:2001. **In this work we demonstrate and discuss how we calculate the detection limit for the determination of ^{210}Pb by liquid scintillation counter (LSC) for the previously developed methodology and compare its results with the MDA value. The DL value found was 0.033 Bq/L instead of 0.032 Bq/L for MDA. The MDA and LD values did not differ virtually, the results highlight that the main change between the two Standard is related to the definition approach.**

Keywords: **detection limit, ^{210}Pb, LSC.**

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1. INTRODUCTION

In 2010 the International Organization for Standardization (ISO) abandoned the concept of minimum detectable amount (MDA) used in ISO 12790-1:2001 [1] and adopted the detection limit (DL) presented in ISO 11929:2010 [2]. In the same way, ISO 28218:2010 “Performance criteria for Radiobioassay” [3] utilizes the concept of detection limit introduced by ISO 11929:2010 in its content. In 2013, the In Vitro Bioassay Laboratory (LBIOVT) from the Dosimetry Division (DIDOS) of the Institute of Radioprotection and Dosimetry (IRD) developed a new approach for the determination of ^{210}Pb by liquid scintillation counter (LSC) [4]. In that paper [4], LBIOVT/DIDOS/IRD presented the DL values found for the methodology developed according to ISO 28218:2010. This work will demonstrate and discuss how the detection limits were calculated for that methodology and compare its results with the MDA.

2. MATERIALS AND METHODS

2.1. General

The standard ISO 28218:2010 “Performance critera for Radiobioassay” [3] has all the necessary formulas to help calculate the detection limit and even estimate the uncertainty, which was done from the partial derivatives according to ISOGUM [5].

With this, ISO 28218:2010 [3] was used throughout this work as a reference and aid in the execution of calculations, and when necessary, support was sought in ISO 11929:2010 [4] and ISO GUM [5].

The value of DL indicates the ability of a laboratory to detect a radionuclide in a sample and is defined as “smallest true value of the measurand that is detectable by the measuring method” by ISO 28218:2010 [3]. ISO 28218:2010 presents an equation model in ionizing-radiation measurements that simplifies all the subsequent calculations.

2.2. The model

According to ISO 28218:2010 [3] the equation model for general chemical analytics can be specified as:

$$y = (x_1 - x_2).w \quad (1)$$

It is important to note that x_1 and x_2 values correspond to the gross count of the sample and the background count respectively. The other input qualities, x_i, are related to the methodology and refer to the efficiency, yield, volume or mass, corrections etc. They are represented by *w*.

$$w = \frac{(x_6.x_8 \ldots)}{(x_5.x_7 \ldots)} \quad (2)$$

The model for determinations of ^{210}Pb by LSC according to the methodology developed is:

$$A = \frac{n_S - n_0}{t.\varepsilon.Q.Y} \quad (3)$$

Where:

A	activity measured in the sample;
n_S	gross counts of the sample;
n_0	gross counts of the appropriated blank;
t	sample counting time;
ε	counting efficiency value for the given time after the precipitation;
Q	quantity of the sample measured;
Y	chemical yield.

Based on equations (1) and (2):

$$y = A;\ x_1 = n_S;\ x_2 = n_0; x_5 = t; x_6 = 1; x_7 = \varepsilon; x_9 = Q; x_{11} = Y \quad (4)$$

$$\boldsymbol{A = (n_S - n_0).w} \quad (5)$$

$$\boldsymbol{w = 1/(t.\varepsilon.Q.Y)} \quad (6)$$

2.3. The uncertainties

The standard uncertainty u(y) is as follow:

$$u^2(y) = w^2.[u^2(x_1) + u^2(x_2)] + y^2 u_{rel}^2(w) \quad (7)$$

Equation (3) is divided into three parts: the uncertainty contributions from the sample, from the background (both being uncertainty type *a*) and the other uncertainty contributions (type *b*), e.g. efficiency, yield etc.

All the uncertainty sources related to *w* are represented by $u_{rel}(w)$ and can be calculated as:

$$u_{rel}^2(w) = \sum_{i=5}^{m} \frac{u^2(x_{i)}}{x_i^2}, i \geq 5 \tag{8}$$

In case of using count rates (x_r) the following relation can be used:

$$x_r = counts/time = n/t \tag{9}$$

$$u^2(x_r) = n/t^2 \tag{10}$$

Setting the uncertainties:

$$u^2(x_1) = n_S; u^2(x_2) = n_0 \tag{11}$$

$$u^2(x_5) = 0; u^2(x_6) = 0; u^2(x_7) = u^2(\varepsilon); u^2(x_9) = u^2(Q); u^2(x_{11}) = u^2(Y) \tag{12}$$

Based on equation (7) and (8) and considering the sample counting time the same as the background counting time ($t_S = t_0 = t$):

$$\boldsymbol{u^2(A) = w^2(n_S + n_0) + A^2 u_{rel}^2(w)} \tag{13}$$

$$\boldsymbol{u_{rel}^2(w) = \frac{u^2(\varepsilon)}{\varepsilon^2} + \frac{u^2(Q)}{Q^2} + \frac{u^2(Y)}{Y^2}} \tag{14}$$

By replacing y with $\tilde{y}$ (true value of the measurand), equation (1) is solved for x_1:

$$\tilde{y} = (x_1 - x_2).w \rightarrow x_1 = \frac{\tilde{y}}{w} + x_2 \tag{15}$$

And based on equation (5) and (6), equation (15) can be rewritten as:

$$n_S = \frac{\tilde{A}}{w} + n_0 \tag{16}$$

By replacing n_S from equation (16) on equation (13):

$$u^2(\tilde{A}) = \left[w^2\left(\frac{\tilde{A}}{w} + 2n_0\right)\right] + \tilde{A}^2 u_{rel}^2(w) \tag{17}$$

2.4. Decision threshold $\boldsymbol{y}^*$

DL value is associated with the decision threshold value, which is a fixed value corresponding to the quantification of the presence of the physical effect. Then firstly, the decision threshold shall be calculated to determine if the count rate from the measurand under analysis is different from the count rate of the appropriate blank.

The decision threshold is calculated as:

$$y^* = k_{1-\alpha}.\tilde{u}(0) \tag{18}$$

Where:

α the probability of wrongly rejecting the hypothesis (error of the first kind);

$\tilde{u}(0)$ uncertainty of the true value.

Considering that the decision threshold is the ability to quantify the physical phenomenon, then assuming that the true value is zero ($\tilde{A} = 0$) in equation (17):

$$\tilde{u}^2(0) = w^2 2n_0 \rightarrow \tilde{u}(0) = w\sqrt{2n_0} \tag{19}$$

And applying equation (19) on (18):

$$\boldsymbol{y^* = k_{1-\alpha}.w.\sqrt{2n_0}} \tag{20}$$

2.5. Detection limit $y^{\#}$

The detection limit $y^{\#}$ is the smallest solution of Equation (21):

$$y^{\#} = y^* + k_{1-\beta}.\tilde{u}(y^{\#}) \tag{21}$$

Being β the probability of wrongly not rejecting the hypothesis (error of the second kind).

According to ISO 28218:2010, the detection limit can be calculated as:

$$\boldsymbol{y^{\#} = \frac{k.w.\left(k+2\sqrt{2n_0}\right)}{1-\left[k^2 u_{rel}^2(w)\right]}} \tag{22}$$

Assuming the probability of the error of first kind being equal to the probability of error of second kind ($k_\alpha = k_\beta = k$).

2.6. MDA calculation

The minimum detectable activity can be calculated as (L.Currie) [6]:

$$\boldsymbol{MDA = \frac{3+4.65\sqrt{n_0}}{t.\varepsilon.Q.Y}} \tag{23}$$

3. RESULTS AND DISCUSSION

3.1. Equations

The equations utilized in this work were:

Equations (5) and (6) for the model:

$$A = (n_S - n_0).w \tag{5}$$

$$w = 1/(t.\varepsilon.Q.Y) \tag{6}$$

Equations (13) and (14) for uncertainties:

$$u^2(A) = w^2(n_S + n_0) + A^2 u_{rel}^2(w) \tag{13}$$

$$u_{rel}^2(w) = \frac{u^2(\varepsilon)}{\varepsilon^2} + \frac{u^2(Q)}{Q^2} + \frac{u^2(Y)}{Y^2} \tag{14}$$

Equation (20) for decision threshold:

$$y^* = k_{1-\alpha}.w.\sqrt{2n_0} \tag{20}$$

Equations (22) and (23) for DL and MDA respectively:

$$y^\# = \frac{k.w.(k+2\sqrt{2n_0})}{1-[k^2 u_{rel}^2(w)]} \tag{22}$$

$$MDA = \frac{3+4.65\sqrt{n_0}}{t.\varepsilon.Q.Y} \tag{23}$$

3.2. Experimental values and results

Table 1 presents the input data obtained for the determination of ^{210}Pb by LSC with the methodology previously developed by the LBIOVT. These results are from a single intercomparison analysis from a water sample containing from 0.37 Bq/L to 2.22 Bq/L of ^{210}Pb.

Table 1: Input data by LBIOVT for ^{210}Pb determination by LSC.

Quantity	Symbol	Value	Standard uncertainty	Unit
Sample counts	r_S	27.821	0.669	cpm
Background count	r_0	2.915	0.173	cpm
Counting time	t	60	-	min
Counting efficiency	ε	70.90	9	%
Sample aliquot	Q	0.963	0.006	L
Chemical yield	Y	82.5	5	%

Table 2 presents the output data obtained after the calculations done with equations (5), (6), (13), (14), (20), (22) and (23), considering the probability of the error of first kind and second kind equal to 5% ($k = 1{,}645$).

Table 2: Results according to the equations presented in this paper.

Quantity	Symbol	Value	Unit
Activity	A	0.737	Bq/L
Standard uncertainty	$u(A)$	0.079	Bq/L
Decision threshold	y^*	0.015	Bq/L
Detection Limit (DL)	y^*	0.033	Bq/L
Minimum detectable activity	MDA	0.032	Bq/L

4. CONCLUSION

We emphasize that this considers only one methodology - ^{210}Pb by LSC - other methodologies, such as ^{226}Ra by LSC, are being evaluated.

MDA and LD values did not differ virtually. For the evaluated method, the new concept does not impact the results and for internal dosimetry there is no difference between MDA and DL. These results stress that the main change between the two standards, ISO 12790-1:2001 and ISO 11929:2010, is related to the definition approach. We reinforce that the LD calculation considers the contribution of variables other than background, unlike the MDA, and that its value changes with each determination. The result obtained refers to the application of the equations described in the article, without any attempt to experimentally evaluate the value presented in table 02 (0.033 Bq/l).

ISO 11929:2010 brings, in addition to the concept of DL instead of MDA, the concept of decision threshold: the value that quantifies the physical effect.

Even so, LBIOVT/DIDOS/IRD is working on other measurements that may bring more understanding about this change along with the ISO Standards.

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