Comparison between three weighing methods for source preparation in radionuclide metrology

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Abstract. In radionuclide metrology three weighing methods could be used for source preparation. The Pycnometer, Elimination and Modified Elimination methods should be compared to evaluate the compatibility in the deposited mass results, so increasing their reliability. In this work, a sequence of micro drop deposition weighing is presented which allows comparing the results by the same drop deposition. The results from each method were determined and their compatibility was checked. As a result, the three methods based on carefully weighing were found compatible and comply with the required threshold uncertainty. Furthermore, it was confirmed the results are traceable to mass the mass unit.

Keywords. Radionuclide metrology, source preparation, weighing methods, uncertainty evaluation.

1. Introduction
In radionuclide metrology, radioactive sources preparation encompasses a weighing procedure able to achieve standard uncertainties below 20 µg in the range from 10 mg to 100 mg. In order to perform this task three weighing methods could be applied: the Pycnometer, the Elimination and the Modified Elimination methods. All of them meet the requirement for micro-drops deposition or dilution of a master solution using a plastic pycnometer [1] provided that they are properly applied thus avoiding doubts about source preparation results [2].

The Pycnometer method corresponds to the differential weighing of the plastic pycnometer before and after drop deposition [3]. In Elimination method [4] three weighing steps are performed per source: the pycnometer is weighed before and after dispensing the drop of solution, and by knowing the mass of the drop (by the weighing difference), one or several standard weights [5] are added on the balance load receptor and the third weighing reading is recorded. The presence of systematic errors in weighing (evaporation, drying of a drop in capillary stem, zero drift of the balance) is checked by the criterion that the difference between third and second weighing readings should be in agreement with conventional mass [6] of standards within twice the uncertainty. As in Elimination, in the Modified version three weighing are performed, but the two last one are performed by using mass standards, whose mass is previously estimated by the known of the quantity of drops deposited and the mass of
one drop (quantitative deposition). Different from the former the check for errors in weighing is executed by the repeatability comparison against historical data [7].

Both types of Elimination methods require the sum of mass standards to be adjusted in the balance load receptor until the difference to first reading be less than about 3 mg (for 0.001 mg resolution balance), so limiting non-linearity error [8]. Then, they are more complex to execute in practice than Pycnometer method. On other side, in order to properly correct the non linearity in weighing by Pycnometer method it is required to evaluate it periodically in a systematic way by calibration using mass standards. Furthermore, the Elimination methods provide some check for errors so they are regarded more reliable than Pycnometer. However, besides the check results, the confirmation about errors existence has to be based on the observation of events in weighing and in the technical experience which should support it. By this way, even without any check procedure, a carefully weighing by an experienced technician using Pycnometer method makes it as reliable as Elimination. Considering the possibility of using any of the three methods, it is important to compare its weighing results in order to evaluate the compatibility of them.

Thus, in this study are presented the results of the comparison between Pycnometer, Elimination and Modified Elimination methods in determining the mass of deposited micro drops. A sequence of weighing results by each method from the same drop deposition was performed. These results in conjunction with effects on weighing were introduced in the mass measurement models. The uncertainty evaluation is based on ISO GUM approach [9] and the compatibility is evaluated by the weighted mean approach [10].

2. Measurement model and uncertainty evaluation

Figure 1 shows the effects on a differential weighing result $\Delta w$ to determine a mass $m$ for a deposited micro drop in an electronic balance. These effects are based on the specified at Euramet Guide 18 [11] in conjunction of [4, 12–14] and it will be detailed in next sections. However two effects are introduced here, the stability of repeatability $\delta_{Stab}$ [15] and non linearity error $\delta_{EStab}$ which are considered to preventively account for changes of balance properties between intermediate checks or calibrations [16].

From the effects, the measurement model for the weighing result of Pycnometer method is (1).

$$\Delta w = R + \delta_{TS} + \delta_{ST} + \delta_{B} + \delta_{Evap} - \delta_{E} - \delta_{EStab}$$

The measurement model for Elimination method includes the standard weight mass $m_{std}$ and the mass stability $\delta_{std}$ (2) and eliminates the balance error effects.

$$\Delta w = R + m_{std} + \delta_{std} + \delta_{TS} + \delta_{ST} + \delta_{B} + \delta_{Evap}$$

Following the nomenclature used in the Euramet Guide, $R$ is the difference between indications $R$, obtained from the respective method. The measurement model for $R$ is given in (3), and includes components that depend only on balance characteristics.

$$R = R_{L} + \delta_{R_{l}} + \delta_{R_{j}} + \delta_{A} + \delta_{X} + \delta_{Stab}$$

Then, the mass of the drop measurement model can be established (4).

$$m = \Delta w \times Bu$$
The buoyancy effect term $B_u$, in Guide, is determined from air density $\rho_a$, radionuclide solution density $\rho_s$ and the conventional density of a standard weight $\rho_c$ [6], (5).

$$B_u = 1 + \rho_a \left( 1/\rho_s - 1/\rho_c \right)$$

The air density $\rho_a$ is calculated from temperature $T$ (°C), pressure $P$ (Pa) and relative humidity $RH$ (%) (6).

$$\rho_a = \frac{0.34848P - 0.009RH \exp(0.061T)}{273.15 + T}$$

As GUM’s principle, each parameter is an input variable in mass measurement model and its standard uncertainty contributes to uncertainty of mass measurement.

Figure 1 Cause-and-effect diagram for mass result of the drop based on effects in weighing on electronic analytical balances.

2.1. Uncertainty on weighing result $u(\Delta w)$

2.1.1. Repeatability ($\delta_r$). This effect accounts for the random variation the difference between indications $R_L$. The effect value is considered null but its uncertainty $u_\Delta$ is estimated from mean of the historical standard deviations obtained in repeatability checks to $R_L$. In Modified Elimination method it is possible to estimate this uncertainty component from the two repeated weighing.

2.1.2. Stability of Repeatability ($\delta_{stab} \Delta$). The repeatability of the method at the weighing time could be different from that obtained as the mean value of previous intermediate checks. This effect is expected to be null in mean in the uniform range of +/- the absolute value of the maximum change in standard deviation from the typical standard deviation $\Delta s$ obtained in the historical series of repeatability checks (7).
\[ u_{Stab} = \frac{\max(\Delta s)}{\sqrt{3}} \] (7)

2.1.3. Resolution (\(\delta_R\)). It corresponds to the rounding error in the last digit of the balance display, estimated by zero within the range of +/- half the scale resolution (\(d_0\)).

\[ u_R = \frac{d_0}{2} \frac{1}{\sqrt{3}} \] (8)

2.1.4. Thermal sensitivity variation (\(\delta_{TS}\)). This effect concerns the variation of the balance sensitivity with the ambient temperature due to the thermal expansion effects on parts of the balance and the magnetization variation in the electromagnetic compensation cell. This effect applies only to weighing difference \(R_L\), its expected value is considered null, but it is evenly distributed over the range of +/- half of \(R_L K_T \Delta T\), where \(K_T\) is the coefficient of variation of sensitivity with temperature obtained from the manufacturer's manual and \(\Delta T\) is the typical variation of ambient temperature in weighing (9).

\[ u_{TS} = R_L \frac{(K_T \Delta T)}{2} \frac{1}{\sqrt{3}} \] (9)

2.1.5. Temporal stability of balance sensitivity (\(\delta_{ST}\)). It is the drift effect of the balance sensitivity or adjustment since its calibration, when the balance is not adjusted before weighing. When the balance is adjusted before weighing, this effect represents the reproducibility of the adjustment. This effect applies to the difference \(R_L\) and its numerical value is null, but is evenly distributed in the range of +/- \(|\Delta E_{max}|/\max\). \(|\Delta E_{max}|\) is the maximum permissible balance error according to document OIML-R76 [17] and \(\max\) is the maximum balance capacity (10).

\[ u_{ST} = R_L \frac{|\Delta E_{max}|/\max}{\sqrt{3}} \] (10)

2.1.6. Buoyancy effect on balance adjustment (\(\delta_B\)). This component is due to variations in air density between the last balance adjustment and weighing time and applies to the difference \(R_L\). The most conservative approach is to assume this effect null within the range of +/- \(R_L 0.1 \rho_0/\rho_c\). Where, \(\rho_0 = 1.2 \text{ kg m}^{-3}\) and \(\rho_c = 8000 \text{ kg m}^{-3}\) are, respectively, the conventional air density and the conventional density of a standard weight (11).

\[ u_B = R_L \frac{0.1 \rho_0/\rho_c}{\sqrt{3}} \] (11)

2.1.7. Eccentricity (\(\delta_X\)). Although weighing is carried out in the center of the balance load receptor, this component is conservatively considered. The effect depends on \(R_L\) and is supposed to be null in the range of +/- \(R_L \left| \Delta L_x \right|_{max}/L_x\). \(|\Delta L_x|\) is the maximum eccentricity indication obtained in the eccentricity check (for example, in balance calibration) and \(L_x\) is the eccentricity measurement point (12).
2.1.8. Evaporation ($\delta_{\text{Evap}}$). Pycnometer evaporation effect was established by measurement as 0.3 $\mu$g/min. It is conservatively assumed that when preparing a source, which takes about 1 min, the effect is null, but varies randomly with standard deviation $u_{\text{Evap}}$ of 1 $\mu$g.

2.1.9. Non linearity error ($\delta_{E}$). This effect is avoided in Elimination methods, but not in Pycnometer. The most accurate way to determine it is performing a calibration in the range 10 mg to 200 mg for a load about the mass of pycnometer full. We have calibrated the balance with calibrated weights and the stability of them was accounted for. In mean the error is null and the uncertainty of this determination is 5 $\mu$g.

2.1.10. Stability of non linearity error ($\delta_{E\text{Stab}}$). This effect accounts to possible variations of the error between calibrations and intermediate checks. It is considered null in the uniform range +/- half $|\Delta E|$ max. $|\Delta E|$ max is the maximum change of the errors obtained from calibrations and intermediate checks, 27 $\mu$g in this work.

2.1.11. Standard weight mass ($m_{\text{Std}}$). The mass of the standard weights is known from its calibration report which provides too the standard uncertainty as a range of possible numerical mass values which could occur due to the process randomness.

2.1.12. Stability of Standard weight mass ($\delta_{\text{Std}}$). This effect accounts for the mass drift from the standard weight usage between calibrations and intermediate checks. It is considered null but is uniformly distributed in the range of +/- $U$, the expanded uncertainty of the standard weights.

2.1.13. Standard uncertainty of the weighing result ($\Delta w$). The standard uncertainty of the difference $R$ is determined by applying GUM’s principle to (3) as in (13).

$$ u(R) = \sqrt{2u_R^2 + u_A^2 + u_X^2 + u_{\text{Stab}}^2} \quad (13) $$

The standard uncertainty for the weighing result $\Delta w$ comes from (1) and (2), respectively for Pycnometer (14) and Elimination methods (15).

$$ u(\Delta w) = \sqrt{u^2(R) + u_{TS}^2 + u_{ST}^2 + u_B^2 + u_{\text{Evap}}^2 + u_E^2 + u_{E\text{Stab}}^2} \quad (14) $$

$$ u(\Delta w) = \sqrt{u^2(R) + u_{TS}^2 + u_{ST}^2 + u_B^2 + u_{\text{Evap}}^2 + u_{m\text{Std}}^2 + u_{\text{Std}}^2} \quad (15) $$

2.2. Buoyancy effect uncertainty $u(Bu)$

2.2.1. Air density uncertainty $u(\rho_a)$. As in (6), air density uncertainty relies on uncertainties of environmental parameters. Besides of these uncertainties components, there is an associated
uncertainty to equation (6), \( u_{\text{form}} = 2.4 \times 10^{-4} \). These parameters contribute to uncertainty of air density (16). The sensitivity coefficients for pressure, relative humidity (in %) and temperature are, respectively \( a_P = 1 \times 10^{-5} \text{ Pa}^{-1} \), \( a_{\text{RH}} = 9 \times 10^{-3} \) and \( a_T = 4 \times 10^{-3} \text{ K}^{-1} \).

\[
\begin{align*}
  u(\rho_a) = \rho_a \sqrt{a_P^2 u^2(P) + a_{\text{RH}}^2 u^2(RH) + a_T^2 u^2(T) + u_{\text{form}}^2} \\

de (16)
\end{align*}
\]

2.2.2. Pressure uncertainty \( u(P) \). According to the Guide the local pressure can be determined according to the equation (17):

\[
P = 1013.25 \exp(-h \times 0.00012)
\]

This equation provides the local atmospheric pressure value in hPa. In this equation \( h \) is the height of the balance above sea level, the value of \( h = 1 \text{ m} \) includes the height of the bench relative to the ground, since the LNMRI is at sea level. The average laboratory pressure has a value of 1014 hPa in a measured range of 1000 hPa \( \leq P \leq 1030 \text{ hPa} \). The pressure measurement is regarded normally distributed with standard uncertainty of \( u(P) = 16 \text{ hPa} \).

2.2.3. Temperature uncertainty \( u(T) \). The temperature during weighing is measured and the working average is 20 °C. The range of maximum variation during weighing is 19°C \( \leq T \leq 21 \text{ °C} \). Temperature is considered uniformly distributed about the measurement temperature in the range of +/- half of 2°C.

2.2.4. Relative humidity uncertainty \( u(\text{RH}) \). The relative humidity during weighing is measured and its uncertainty is assumed to be, in a conservative way, from that uniformly distributed with working average of 67.5% in the middle of the symmetric range of 55 % \( \leq \text{RH} \leq 80 \% \).

2.2.5. Radionuclide solution density \( \rho_s \). Radionuclide solutions are mostly based on aqueous solutions so the density is considered uniformly distributed in the half of the symmetric interval about the mean \( 1.0 \text{ g cm}^{-3} \leq \rho_s \leq 1.1 \text{ g cm}^{-3} \).

From air and solution densities uncertainties, respectively, \( u(\rho_a) \) and \( u(\rho_s) \) it is possible to determine de standard uncertainty of the buoyancy effect (18).

\[
\begin{align*}
  u(\text{Bu}) = \Delta w \sqrt{u_a^2 \left( \frac{1}{\rho_s} - 1/\rho_c \right)^2 + \rho_a^2 u_s^2 \frac{\rho_s^4}{\rho_s^4}} \\

de (18)
\end{align*}
\]

2.3. Deposited mass uncertainty
From the model of mass measurement in equation (4) the uncertainty for mass of the deposited drop is (19).

\[
\begin{align*}
  u(m) = \sqrt{u^2(\Delta w) + u^2(\text{Bu})} \\

de (19)
\end{align*}
\]

3. Weighing procedures
In this study, drop depositions were performed in a routinely way by using a polyethylene pycnometer. The weighing was performed in a 52 g full range microbalance properly chosen [18]. The environmental conditions were recorded by a temperature, relative humidity and pressure sensor and
the data was collected by a Labview application from an Arduino nano board. Due to most of radionuclide solutions are aqueous and following Alara’s principle, the weighing was executed using distilled water. A fifteen minutes wait time was used for thermal stabilization between balance, pycnometer, environment and technician.

Drop deposition is a non repetitive task (unique event), because the limited control in pressing force that even an experienced weighing operator has. Under repeatability conditions (same weighing operator, environmental condition, balance and pycnometer) the repeatability in drop deposition is about 6% for a drop mass deposited in the range 10 mg to 200 mg, so it is higher than the specified uncertainty limit of 20 $\mu$g required for radionuclide source preparation. Thus, in order to measure the same mass deposited applying the three different methods a sequence of four weighing was developed considering the common weighing between the three methods, Figure 2.

![Figure 2 Weighing sequence: four weighing from only one drop deposition.](image)

In the sequence: a) the first weighing is performed with a full pycnometer and a balance reading is recorded $I_1$, b) the second is after drop deposition, reading $I_2$, c) the third is performed putting standard weights until the difference $I_3 - I_2$ is about 3 mg and d) the last one is performed after removing and replacing the pycnometer and the standard weights. From the weighing sequence difference between indications $R$ is obtained (20-22), respectively to Pycnometer (P), Elimination(E) and Modified Elimination (ME) methods.

\[
R_{LP} = I_1 - I_2 \tag{20}
\]

\[
R_{LE} = I_1 - I_3 \tag{21}
\]

\[
R_{LME} = I_1 - \frac{I_3 + I_4}{2} \tag{22}
\]

Each individual balance reading shows some randomness, accounted for repeatability which varies in function of reading. In case of both elimination methods where the individual readings are the same magnitude the same typical repeatability $\sigma$ can be attributed to them. Thus the expected repeatability
of Elimination method $\sqrt{2} \times \sigma$ is little higher than in Modified Elimination $\sqrt{3}/\sqrt{2} \times \sigma$. Furthermore, due to the two repeated readings, $I_3$ and $I_4$ in (22), it is possible to estimate $\sigma$. In this way, the estimated repeatability can be used in place of that obtained from intermediate checks since multiplied by the factor $t^{\alpha}(1; 68.27\%) \times \sqrt{3}/\sqrt{2} \approx 2.25$ and when the multiplied repeatability is smaller than the historical one. The first term in the factor comes from the confidence interval to estimate repeatability and it is the quantile of t-distribution for 1 degree of freedom and a coverage probability of 68.27%.

4. Results

Table 1 shows input data obtained for the eight weighing series performed in an electronic balance Mettler Toledo XP52. The environmental conditions during weighing were $T = 19.0(0.6)$ °C, $RH = 60.0(7.2)\%$, $P = 1014(16)$ hPa resulting in an air density $1.21(0.02)$ kg m$^{-3}$.

| $I_1$  | $I_2$  | $I_3$  | $I_4$  | $m_{std}$ | $u_{mstd}$ |
|-------|-------|-------|-------|-----------|------------|
| 3398.445 | 3374.228 | 3393.906 | 3393.906 | 19.6732 | 0.0015 |
| 3428.561 | 3410.686 | 3430.359 | 3430.369 | 19.6732 | 0.0015 |
| 3319.494 | 3293.126 | 3312.794 | 3312.794 | 19.6732 | 0.0015 |
| 3308.434 | 3272.86 | 3292.536 | 3292.536 | 19.6732 | 0.0015 |
| 3290.278 | 3279.235 | 3289.221 | 3289.221 | 9.9856 | 0.0015 |
| 3558.546 | 3536.914 | 3556.909 | 3556.909 | 19.9970 | 0.0015 |
| 3353.494 | 3113.683 | 3353.673 | 3353.665 | 239.9734 | 0.0037 |
| 3775.687 | 3709.477 | 3775.274 | 3775.274 | 65.7874 | 0.0029 |

Additional parameters required for uncertainty calculation are shown in Table 2.

| Quantity                  | Parameters       |
|---------------------------|------------------|
| Typic. Repeat. Mod. Elim  | $8 \mu g$        |
| Resolution                | $d_0 = 1 \mu g$  |
| Eccentricity              | $| \Delta I_x |_{max} = 32 \mu g$ |
|                           | $L_x = 20 g$     |
| Sensitivity Temp          | $K_T = 1 \times 10^{-6} °C^{-1}$ |
|                           | $\Delta T = 2 °C$ |
| Stab. Sensitivity         | $| \Delta E_{max} | = 1.5 mg$ |
|                           | max = 52 g       |
| Adj. Buoyancy             | $\rho_0 = 1.2 kg m^{-3}$ |
|                           | $\rho_c = 8000 kg m^{-3}$ |
| Evaporation               | 1 $\mu g$       |
| Non Linearity uncert.     | 5 $\mu g$       |
|                           | $| \Delta E |_{max} = 27 \mu g$ |

As an example, the uncertainty budget to the first measurement for each method is shown in Table 3. In this example, the Modified Elimination method reaches the lower uncertainty among the three methods. When repeatability in loco is lower than the historical value it replaces the last one.

Table 4 presents the deposited mass results for each method and the weighted mean analysis values.
| Table 3: Uncertainty budget. |
|-----------------------------|
| **Quantity** | **Pycnometer** | **Elimination** | **Modif. Elimination** |
|----------------------------|----------------|----------------|-----------------------|
| Value u(mg) | Value u(mg) | Value u(mg) | Value u(mg) |
| Repeatability | 0 0.0050 | 0 0.0080 | 0 0 |
| Stab. Repeatability | 0 0.0020 | 0 0.0050 | 0 0.0050 |
| Resolution | 0 0.0003 | 0 0.0003 | 0 0.0003 |
| Eccentricity | 0 0 | 0 0 | 0 0 |
| **Reading diff. R** | 24.2170 0.0055 | 4.5390 0.0092 | 4.5390 0.0046 |
| Sensitivity Temp | 0 0 | 0 0 | 0 0 |
| Adj. Buoyancy | 0 0.0002 | 0 0 | 0 0 |
| Evaporation | 0 0.0006 | 0 0.0006 | 0 0.0006 |
| Non Linearity | 0 0.0050 | 0 0 | 0 0 |
| Stab. Non Linearity | 0 0.0078 | not applicable | |
| Standard weight | not applicable | 19.6732 0.0015 | 19.6732 0.0015 |
| Stab. Stand. Weight | 0 0.0017 | 0 0.0017 | 0 0.0017 |
| **Weighing result ∆w** | 24.2170 0.0108 | 24.2122 0.0108 | 24.2122 0.0052 |
| Buoyancy | 1.001000 0.000036 | 1.001000 0.000036 | 1.001000 0.000036 |
| Deposited mass | 24.241 0.011 | 24.236 0.010 | 24.236 0.005 |
| Relative uncert. | 0.04% | 0.04% | 0.02% |

| Table 4: Deposited mass and compatibility results. |
|-----------------------------|
| **Mass** | **Pycnometer** | **Elimination** | **Modified Elimination** | **Weighted Mean** | **Maximum Deviation** |
| mass u(mg) | mass u(mg) | mass u(mg) | Value u(mg) | Value |
|-----------------------------|
| 24.241 0.011 | 24.236 0.010 | 24.236 0.005 | 24.237 0.004 | 0.004 0.010 |
| 17.893 0.011 | 17.894 0.010 | 17.889 0.010 | 17.892 0.006 | -0.003 0.008 |
| 26.394 0.011 | 26.400 0.010 | 26.400 0.005 | 26.399 0.004 | -0.004 0.010 |
| 35.610 0.011 | 35.607 0.010 | 35.607 0.005 | 35.607 0.004 | 0.002 0.010 |
| 11.054 0.011 | 11.054 0.010 | 11.054 0.005 | 11.054 0.004 | 0.000 0.010 |
| 21.654 0.011 | 21.656 0.010 | 21.656 0.005 | 21.655 0.004 | -0.002 0.010 |
| 240.051 0.014 | 240.034 0.014 | 240.038 0.014 | 240.041 0.008 | -0.007 0.018 |
| 66.276 0.011 | 66.267 0.011 | 66.267 0.007 | 66.269 0.005 | 0.008 0.010 |

Generally, the uncertainty increases from Modified Elimination to Pycnometer method which presents uncertainty about that of the Elimination method. For higher mass Pycnometer and Elimination provide the same uncertainty because the increase in components which depends on reading difference in the first is balanced by equivalent increase in standard weight uncertainty. For the three methods the maximum relative uncertainty occurred at the lower mass value and the minimum occurred at the higher mass. All of maximum deviations in weighted mean analysis were lower their uncertainty.

5. Conclusion
In this work three weighing methods currently used in radionuclide metrology were compared in order to evaluate the compatibility between them. The results have shown high compatibility between Pycnometer, Elimination and Modified Elimination method provided that the weighing be carefully performed.

Furthermore, the uncertainty results provided information about the accuracy for each method. The lower deposited mass the higher relative uncertainty, a consequence of the closeness to the kilogram.
(lowest relative uncertainty in mass) as one should hope. Thus, the traceability chain from the three methods results to the mass unit is validated.

In the mass range from 10 mg to 200 mg, the uncertainty was lower than the Campion’s threshold 20 μg, indicating that the three methods are suitable to the nowadays employment in radionuclide metrology.

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