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Traceability concept teaching by means of experimental procedures for chemistry undergraduate students at UNAM.

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1. Introduction

The Chemistry Faculty of the Universidad Nacional Autónoma de México, in the previous studies that it realized to update and modify its plans and programs of study, that developed in the actual plans of 2005, takes notice that:

1.1- The metrology has acquired great importance for industry and in consequence for international commerce; in regard to the fact that, in general, the productive systems use processes that demand a more significant control in the uncertainty of measurement, which influences strongly the productions quality and knowing that the pharmaceutical industry (in order to hold higher standards of quality, given the nature of the products that it elaborates), has adopted the necessary actions to guarantee that its products fulfill the rules established by the public health authorities.

1.2.- On a particular matter, if the production follows its own procedures, this must be validated according to its appliance, specificity accuracy and intermediate precision must be determined, in terms of it repeatability. It must be guaranteed that all measurement instruments used in pharmaceutical industry fit the rules of the actual calibration system.

1.3.- On the other hand in the industrial production as well as in the scientific research, exists the obvious necessity of obtaining measurement results with a smaller uncertainty.

The Chemistry Faculty considered convenient to include in the Study plans of the Chemistry career, with a compulsory character, the “Metrology” subject.

This paper presents the teaching experience results of the Metrology lab whose objective is that the students realize the importance of the traceability chain in the measurements they make through the following practical problem:

“You work for the pharmaceutical labs “X” and you must make an experimental job that consists of determinate the average mass of a sample of medical pills for which you must create a report that shows the results obtained. The results you get will be the reason for the production of the pills to be developed or stopped by the health authorities of your country. The criteria adopted by authorities is: To accept the sample, the average mass of pills must be m = 0,2117 $\pm$ 1,0% g"

The work team must obtain results with expanded uncertainty with a level of reliability of 95,45% (coverage factor K = 2)

2. Material, equipment and instruments.

- Ohaus Balance, model Voyager, Special class; resolution of 0,0001 g and range of 210 g; use interval from 1 mg to 210 g.
- Fisher Standard mass set I, class of accuracy M2 with internal code VAL003 calibrated by the National Secondary Lab INSCO that developed the certificate of calibration INF-VAL-1206.
- Mettler-Toledo Standard mass set II, with internal code MAS 007, with use interval from 1 mg to 200 g, class of accuracy F1, the set of mass is calibrated by the "Calibración y Metrología, laboratorio de calibración" lab, wish developed the number report of INF-VAL-1206.
- Cotton gloves that do not lose fibers.
- Thermometer.
- Hygrometer.
- Barometer.
- Clamps covered with Teflon.
- Clothe for clearing the balances.
- Sample of fifty medical pills.

The work team must do the experimental procedure in two separate occasions. During the first part of the work they will use the "Standard mass set I" in the calibration process, and in the second part they will use the "Standard mass set II". Finally, they must elaborate a traceability letter.

3. Experimental Procedure.

3.1.- Balance Calibration.
3.2- Mass measurement of pills.

The average mass of the pills sample is obtained using the equation 1

$$X = \frac{\sum_{i=1}^{n} x_i}{n} \qquad (1)$$

Where X_i is the value of the i-esim mass, *n* is the total number of pills, while the standard deviation is obtained with the equation 2.

$$S = \sqrt{\frac{\sum_{i=1}^{n}\left(\overline{X} - X_i\right)^2}{n-1}} \qquad (2)$$

The type A uncertainty (μ_A), from the pills sample is calculated with equation 3.

$$\mu_A = \frac{S}{\sqrt{n}} \qquad (3)$$

Later, the students must take notice of the uncertainty informed in the balance calibration process, that result is known as type B uncertainty (μ_B), this value is expanded to a level of reliability K = 2.

With both data (μ_A y μ_B), and the balance resolution (μ_r), the mass combined uncertainty of the pills is calculated with equation 4.

$$U_C = \sqrt{\left(\mu_A^2 + \left(\frac{\mu_B}{2}\right)^2 + \mu_r^2\right)} \qquad (4)$$

The resolution uncertainty (μ_r), is obtained by equation 5.

$$\mu_r = \frac{\mathrm{Re}\,solucion}{2\sqrt{3}} \qquad (5)$$

The average mass of the pills sample is informed with expanded uncertainty (μ_{exp}), with a coverage factor K= 2.

$$U_{\exp} = KU_C \qquad (6)$$

According to the certificates of calibration provided by the two calibration labs previously quoted, the results for both Standard mass sets are shown on Table 1.

Table 1.

Parameter	Standard mass set I (g)	Standard mass set II (g)
$\bar{X}$	0,2117	0,2117
S	$\pm$ 7,500 x 10^{-3}	$\pm$ 7, 500 x 10^{-3}
μ_A	$\pm$ 9,400 x 10^{-4}	$\pm$ 9,400 x 10^{-4}
μ_B (K=2)	$\pm$ 12,60 x 10^{-4}	$\pm$ 1,900 x 10^{-4}
μ_B (K=1)	$\pm$ 6,250 x 10^{-4}	$\pm$ 0,950 x 10^{-4}
μ_r	$\pm$ 2,886 x 10^{-5}	$\pm$ 2,886 x 10^{-5}
U_C	$\pm$ 11,29 x 10^{-4}	$\pm$ 9,452 x 10^{-4}
U_{exp}	$\pm$ 22,58 x 10^{-4}	$\pm$ 18,90 x 10^{-4}

It is proper to mention that in both cases the uncertainty is informed with a coverage factor K = 2, the uncertainty is obtained by a test of linearity in the balance [2].

4. Traceability Chart [3, 4, 5].

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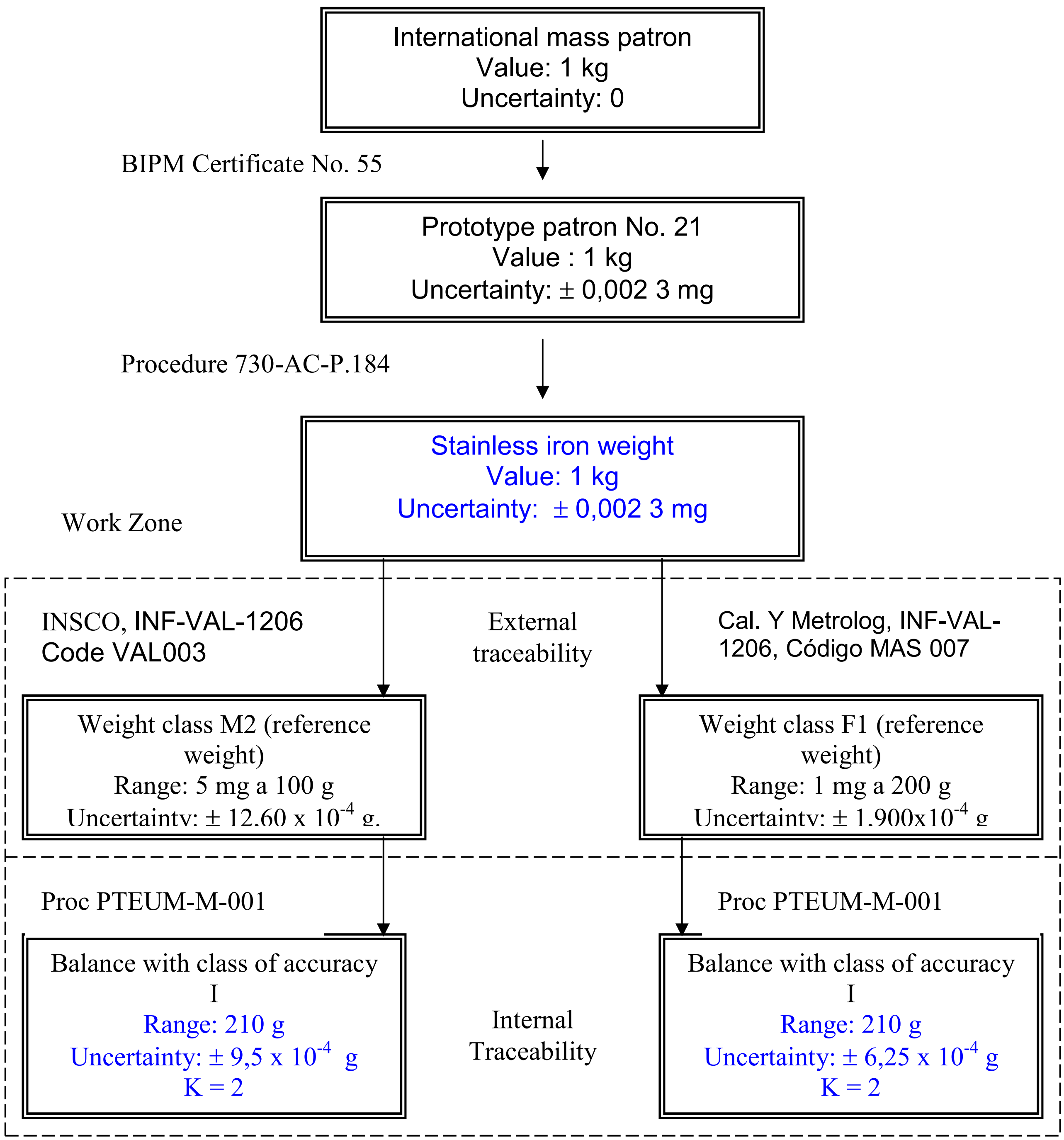
MEASSUREMENTS OF MASS OF A LOT OF PILLS
Method: Simple Weighting



In regard to the traceability, as it may be seen, the results have different uncertainties, by the sole fact of using the same calibrated instrument with two different standard mass sets. Considering that the mass measurement of a medical pills sample follows a normal distribution, the results of the average mass of the pills with its associated uncertainty are illustrated in figure 1.

A. Result using the Standard set of mass I: $X = 0{,}2117 \pm 22{,}58 \times 10^{-4}$ g
B. Result using the Standard set of mass II: $X = 0{,}2117 \pm 18{,}90 \times 10^{-4}$ g

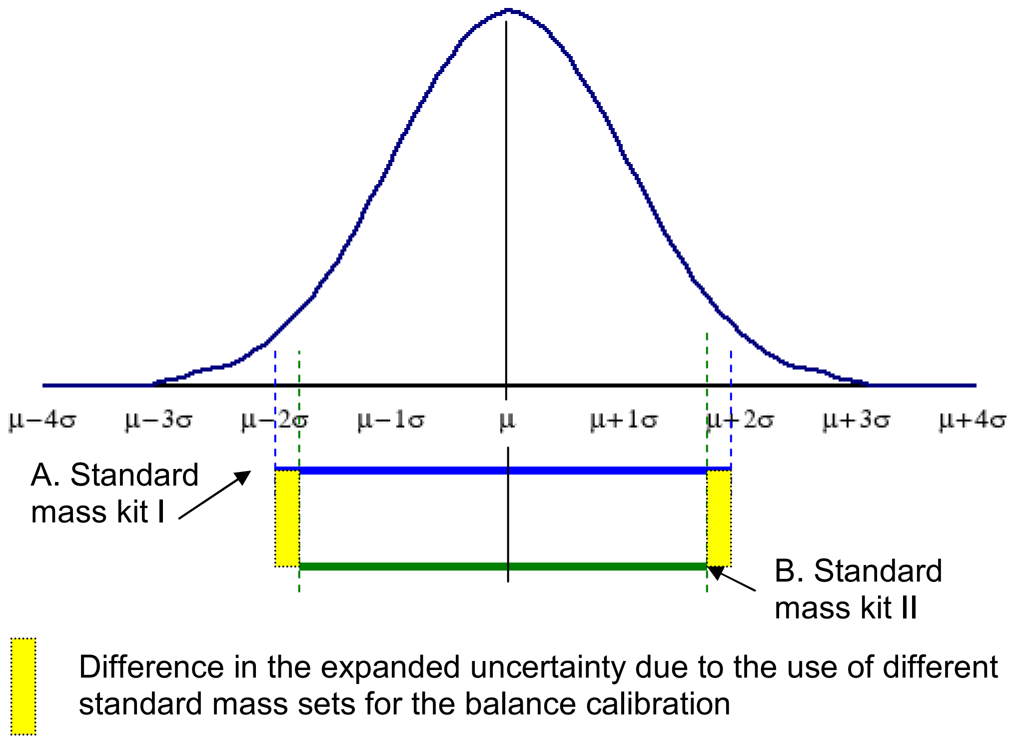


Figure1. Normal distribution curve that shows the result of the mass measurement of a sample of 50 medical pills, the expanded uncertainty is aggregated to a coverage factor K=2.

The difference in the values of the uncertainty due to the calibration correspond to 0,35%.

6. Conclusions.

- Calibrating the balance that we use to determinate the average mass, using different Chains of Traceability, produces different results in the determinations.
- It is evident that the equipment user necessarily must know the importance of adequate interpretation of the calibration certificates content.
- The Traceability letters elaboration contributes in a decisive way to the education of the professionals in Chemistry, as users of the Metrology.

7. References.

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