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Optimized Calibration Plan based on Gray Model GM(1,1) applied in Physical-Photometric and Chemical Laboratories Accredited by ISO/IEC 17025

Carlos Velásquez[1,2,3*], Francisco Espín[3,4], Francisco Iturra[3], Consuelo Chasi[3]

[1]*Dept. of Applied Mathematics, University of Alicante, Apdo. 99, 03080, Alicante, Spain*
[2] *Modalidad en Línea, Universidad Central del Ecuador, Quito 170521 Ecuador*
[3]*Instituto de Investigación Geológico y Energético, Quito 170518, Ecuador*
[4]*Departamento de Luminotecnia, Luz y Visión, Universidad Nacional de Tucumán, San Miguel de Tucumán Tucumán T4000, Argentina*

Abstract. The competence of laboratories to perform testing and calibration services for the industry is assured by the ISO/IEC 17025 accreditation. To comply with this standard, it is imperative to ensure metrological traceability through the regular calibration of laboratory equipment at defined schedules, which is time-consuming and demands significant financial resources. The present work applies first-order gray models GM(1,1) using calibration uncertainties to establish an optimized calibration plan that considers different natural characteristics of laboratory activities, thus providing technical support to ensure metrological traceability for a wide range of laboratory tests. A luminous intensity distribution test on LED luminaires and a fire assay method for determining gold were considered. Applying the proposed approach, the outcoming times between calibrations were longer than previously established, reducing 11% of the financial resources for the physical-photometric laboratory and 54% for the chemical laboratory. Therefore, it also increases the availability of calibrated pieces of equipment. Moreover, results showed that this method suits physical and chemical laboratory tests. In conclusion, this methodology could increase the time between calibrations and reduce the financial resources needed while maintaining technical competence or confidence in laboratory results.

Keywords: Fire assay; Gray model; ISO/IEC 17025 accreditation; Luminous intensity; Uncertainties

1. Introduction

The ISO/IEC 17025:2017 standard *"General requirements for the competence of testing and calibration laboratories*" (ISO, 2017) aims to develop and promote confidence in the operation of laboratories. These standard mandates laboratories to employ calibrated measurement equipment under two conditions: when the accuracy and uncertainty of the measurement affect the validity of the results and when calibration is necessary to establish the metrological traceability of the results. In addition, the standard requires that the laboratory establish a dynamic calibration program to ensure the reliability of the calibration status (ISO, 2017).

There are different ways of carrying out the laboratory calibration program, usually adopted by guidelines suggested by the International Laboratory Accreditation Cooperation

*Corresponding author's email: cavf1@alu.ua.es, Tel.: +593992623890
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and the International Organization of Legal Metrology in ILAC G24 OIML D10 (Legal International Organization of Metrology, 2007). Most laboratories apply year-to-year calibration and instrumental drift calculation methods.

A "dynamic calibration program" changes the paradigm of fixed calibration times. It encourages laboratories to determine, through a series of evaluations, the behavior of the measurement equipment and the most appropriate calibration times (Silva and Rodrigues, 2013). The calibration time can be determined by verifying the behavior of the measuring equipment between calibrations or by estimating the variation of measurement uncertainty over time (Gaber, 2021; Delker, Auden, and Solomon, 2020).

Estimating the calibration time for measurement equipment is a complex issue that involves economic risks when estimating time is too short (Pashnina, 2020) and technical risks when the estimated time is excessively long (Gaber, 2021). An adequate procedure in planning laboratory activities affects the production of its clients (Deepradit, Ongkunaruk, and Pisuchpen, 2020) and its repair times in factories, laboratories, and plans (Wibawa Ichsani, and Yuniarto, 2021). The applicability of gray models is a predominant tool when a laboratory has a history of economic investment made in the calibration of its equipment (Velásquez *et al.,* 2021a).

Various statistical approaches prove useful in determining a calibration interval (Butdee and Khanawapee, 2021; Velásquez *et al.,* 2021b), with extensive research conducted on the topic. Results expose methodologies used to define calibration ranges, among them chain methodology, simple linear model, and decisional model. Additionally, a large number of variables must be considered, such as the maximum permissible errors required by the laboratory, frequency of use of the equipment, type of application, robustness, other requirements determined by manufacturers (Toteva, Slavov, and Vasileva, 2017), and other factors such as laboratory infrastructure (electrical installations, environmental conditions), detect anomalies in equipment (El-Hadad, Tan, and Tan, 2022) or the competence of the personnel operating the equipment (Nugraha *et al.,* 2022).

These models have been generally applied to a single equipment or laboratory. This work presents a new use of gray model study applied to physic and chemical laboratories for its calibration ranges that have not been developed. The first-order gray model GM(1,1) is a good alternative explored in some research. It suggested that due to the characteristic of the technique to project values whose nature of change is unknown (gray action)(Zeng, Ma, and Shi, 2020), it is advantageous to be applied when projecting the changes over time of uncertainties of different laboratory equipment (Lin and Liu, 2005). Applying this methodology allows adjusting calibration intervals considering the reasons for a change of the equipment with adequate predictions.

The model GM(1,1) used in this research needs as a mathematical parameter the calibration uncertainties of equipment involved in the complete test. A calibration certificate usually provides much information to the laboratory, mainly the correction and the uncertainty values (Kopke, Mourão, and Brito, 2024; Taymanov *et al.,* 2023; Delker, Auden, and Solomon, 2020; Muscas *et al.,* 2001). To understand the technical importance of these requirements, it is necessary to consider that all measurement results will always have two essential characteristics: bias and uncertainty. The bias is the difference between the result and true values, while the uncertainty means the dispersion of the quantity values in which the true value could be found with a given confidence level (Velásquez *et al.,* 2024). This information is used to maintain the metrological traceability chain and ultimately determine the technical quality of the delivered results.

The measured value could be adjusted by applying the certificate correction. However, the uncertainty is related to the accuracy and repeatability status of the equipment as well

as the supplier's method of calibration. Through a periodic calibration process of all the equipment whose measurements influence the test results, values of bias and uncertainty are obtained, so that is why the importance of ensuring correct calibrations and their intervals.

Due to their nature, there are physical or chemical laboratories. In both, uncertainty is the fundamental value representing the quality of an assay or a calibration result. Its calculation and application are described through "The Guide to the Expression of Uncertainty in Measurement," also known as GUM (Joint Committee for Guides in Metrology, 2008). It was created by the Joint Committee for Guides in Metrology. It is the best general approach to estimating the uncertainty of analytical processes (González *et al.,* 2018). Moreover, it uses a so-called bottom-to-top method, which has some critical limitations for chemical laboratories where the top-to-bottom approach is more appropriate (Ellison, 2014). As a response to this, the GUM has been adapted by EURACHEM/CITAC in accordance with its guide 'Traceability in chemical measurements' specifically tailored for chemical laboratories (González *et al.,* 2018). The laboratories use, in its experimental scheme for the test, calibrated equipment. The method used by the laboratory has to have a combined uncertainty, in which each of the uncertainties of the calibrated equipment is used.

Even though conceptually, the uncertainty term is always the same, in practice, physical and chemical laboratories have differences in their estimates. However, it is possible to apply an approximation of GM(1,1) regardless of the nature of the activities of the laboratories.

In this work, physical-photometric and chemical-accredited laboratories were the scopes of application (Figure 1a and Figure 1b) (Velásquez *et al.,* 2023). In the case of the physical-photometric laboratory, the assay to determine luminous intensities distribution in LED luminaires employing a goniophotometer was analyzed. On the other hand, for chemical laboratories (Juiña, Silva, and Velásquez, 2024), the analysis focused on the determination of gold concentration in minerals using the 'fire assay' method combined with Atomic Absorption Spectrophotometry.

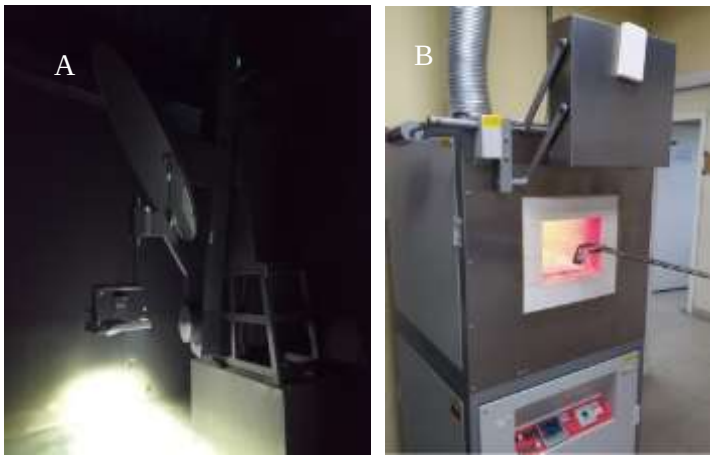

Figure 1 Experiment methods of the assays in physical-photometric laboratory(a) chemical laboratory (b)

2. Methods

The based GM(1,1) methodology proposed was applied in two different assays of different natures: physical-photometric and chemical. It is essential to understand the annual cost of equipment calibration and the traceability chain of each one. The traceability

relationship between two laboratory equipment in the chain (Figure 2, Figure 3) is ζ_i. The ζ_i Represents if there is a traceability transfer.

2.1. Traceability Chain for Physical-Photometric Laboratory

An accredited test in the physical-photometric laboratory measures luminous intensities distribution in LED luminaires using a C-type rotating mirror goniophotometer. The standard methods for the test are CIE 121, "*The Photometry and Goniophotometer of Luminaires*" (CIE, 2009) and CIE S025, "Test Method for LED Lamps, LED Luminaires and LED Modules"(CIE, 2015). In some American countries, IES LM 79 "Optical and Electrical Measurements of Solid State Lighting Products" (Illuminating Engineering Society, 2019), replacing CIE S025 is also common.

The test begins with stabilizing the luminaire by turning it on for two hours at a constant voltage with a power supply regulated at ±0.2%. Next, vector detection is developed through spherical coordinates, where its direction will be given by the axial (C-Planes) and azimuthal (Gamma Angles) angles and its magnitude by the light intensity, describing the photometric volume of interest. Additionally, during the test, a temperature of 25°C ± 1.2°C must be maintained because temperature variation has an impact on the test (Brusil, Espín, and Velásquez, 2021). The environmental conditions of temperature and electrical conditions of voltage, current, and total harmonic distortion (THD) must be supervised (Brusil *et al.,* 2020) during the test.

The measurement of luminous intensities is absolute (cd) for LED luminaires. The calibration of the goniophotometer is done with a work standard lamp. The work standard lamp is traceable to the reference standard lamp with its electrical parameters. The traceability chain is presented in Figure 2.

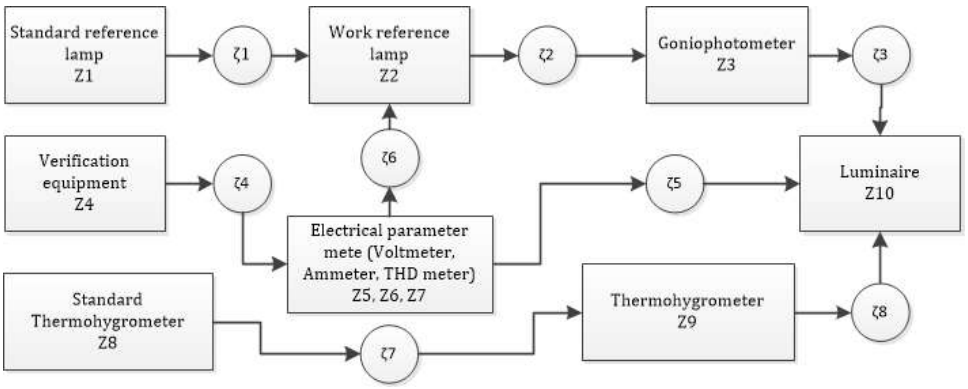

Figure 2 Traceability chain method of the test in the physical-photometric laboratory

The photometric scheme has an incandescent technology standard reference lamp (Z1), an incandescent technology work lamp (Z2), a SENSING GMS-2000 Goniophotometer (Z3), and routine test LED luminaires (Z10). The electrical magnitudes were supervised by a Metrel MI 2892 Electrical Network Analyzer (Z2) and a Yokogawa WT310 Digital Power Meter to detect voltage (Z5), current (Z6), and THDs (Z7). The environmental conditions were measured with a verification equipment Thermohygrometer TESTO 176P1 (Z8) and a routine measurement equipment Thermohygrometer TESTO 174H (Z9).

2.2. Traceability Chain for Chemical Laboratory

The Chemical Laboratory performs mineralogical and elemental tests in various geological matrices. Among other trials, it determines gold by the "fire assay", the reference technique for gold quantification (Buitrón *et al.,* 2021). It is based on the "ASTM E1335, Standard Test Methods for Determination of Gold in Bullion by Fire Assay Cupellation Analysis" and the "3111 Metals by Flame Atomic Absorption Spectrometry". This method is

preferred over others due to its application to a broad spectrum of samples, excellent versatility, and high accuracy (Buitrón *et al.,* 2021). It is also considered that the nugget effect is reduced due to the large amount of sample that can be used.

The equipment used for the assay are as follows: Weights brand Mettler Toledo E2 type (Z1), a scale brand Precisa model XB4200C (Z2), a scale brand Mettler Toledo model XP 205 (Z3), a scale brand Citizen model CX 220 (Z4), a pipette of 500 µL brand Socorex (Z5), a pipette of 5000 µL brand Socorex (Z6), a pipette of 100 µL brand Socorex (Z7), a pipette of 1000 µL brand Socorex (Z8), a dispenser of 10 mL brand Brand (Z9), certified reference material of gold-containing minerals from Rocklabs (Z10), a certified standard of gold from Inorganic Ventures (Z11), an Atomic Absorption Spectrophotometer brand Perkin Elmer (Z12) two Thermo hygrometers brand Traceable (Z13 and Z14), two thermometers brand Fluke (Z15 and Z16), two muffles brand Incinerar (Z17 and Z18), a muffle brand Carbonate (Z19), a stove brand Polenco (Z20) and a hotplate brand SCP Science (Z21).

As is shown in Figure 3, a series of verifications are carried out to ensure the quality of the results. Weights are used to verify scales that later are used to verify the volumetric material (Pipettes and Dispenser). Meanwhile, standard solutions are prepared using pipettes and certified reference material to calibrate and verify an Atomic Absorption Spectrophotometer. Temperature equipment (muffles, hotplate, and stove) is verified by a verified thermometer. In addition, humidity and temperature conditions are monitored using a thermohygrometer.

The samples to be analyzed are dried in an oven at 50 °C. In the first stage of the test, high-temperature equipment is required. The sample is roasted at 700 °C to eliminate interferences; then, the sample is melted with a lead-based flux charge at 1000 °C. Balances are used to prepare the flux charge and weigh the sample. In the second stage, gold is recovered from the lead as a doré through a cupellation process at 950 °C.

Finally, acid digestion of the doré is carried out in a digester at 60 °C, and the resulting solution is analyzed using an atomic absorption spectrometer (Buitrón *et al.,* 2021). By applying the Lambert-Beer law, which relates the concentration of a substance to its absorbance, we can determine the concentration of gold in mg/Kg of the sample. The traceability chain is presented in Figure 3.

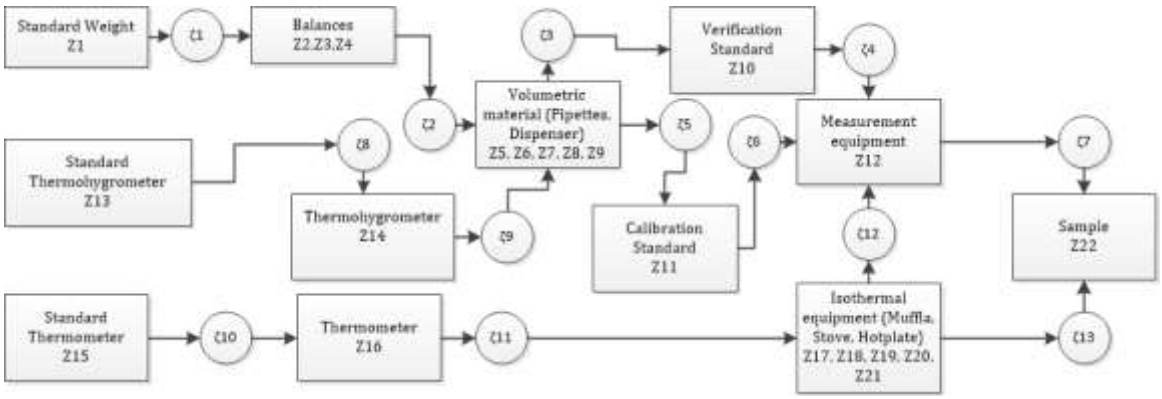

Figure 3 Traceability chain method of the test in the chemical laboratory

2.3. Gray model GM(1,1) applied to uncertainty growth in the function of time

Gray models have an acceptable behavior when it is necessary to model data for which we have incomplete information or to project a value in a trend whose natural causes that would explain its behavior are unknown. The use of the laboratory equipment represents its consumption. In consequence, there must be a loss, no matter how small, of its precision the longer it has been used. This behavior is quantified in the dispersion that occurs when

taking successive measurements. When the equipment is calibrated year after year, it is possible to observe its uncertainty growth as a function of time.

The deterioration of the equipment due to its use has imponderables that may be unknown. For this reason, it is possible to apply GM(1,1) to the modeling of these data. However, the requirements of a test method or a laboratory to comply with its quality standards establish a limit tolerance. We can use this limit as a benchmark to identify the maximum calibration interval of each piece of equipment.

Following reasoning similar to (Wang, Zhang, and Jiang, 2017), it is possible to define Equation 1:

$$\frac{dU^{(1)}}{dt} + aU^{(1)} = b \tag{1}$$

Where $U^{(1)}$ is the uncertainty accumulated of equipment, is the development coefficient, b is the gray action, and t is time.

Using a discretization k for the calibration intervals (in this case, years), the solution is represented in Equation 2 for a vector of uncertainties year a year $Y = \left(U^{(0)}(1), U^{(0)}(2), U^{(0)}(3), \ldots, U^{(0)}(n)\right)$

$$\hat{U}^{(1)}(k+1) = \left(U^{(0)}(1) - \frac{b}{a}\right)e^{-ak} + \frac{b}{a} \tag{2}$$

The predicted uncertainty for period k+1 is given by Equation 3:

$$\hat{U}^{(0)}(k+1) = \hat{U}^{(1)}(k+1) - \hat{U}^{(1)}(k) = (1-e^{a})\left(U^{(0)}(1) - \frac{b}{a}\right)e^{-ak} \tag{3}$$

As Equation 1 and the vector Y show, it is necessary to use the amount n of calibration uncertainty data the laboratory uses to find the constants a and b. With these results and using Equation 3, it is possible to find the projected values and identify the period in which the uncertainty will be outside its tolerance.

3. Results and Discussion

3.1. Implementation algorithm GM(1,1) given a vector of uncertainties Y

The first step to implementing the proposed methodology is to use the following algorithm that will allow the solving of GM(1,1) for each vector Y associated with each piece of equipment present in the different traceability chains Figures 1 and 2:

Step 1: Define the vector Y with its n calibration data

Step 2: Calculates the cumulative vector of uncertainties, $U^{(1)}(k) = \sum_{i=1}^{k} U^{(0)}(i)$, $k = 1,2,\ldots,n$

Step 3: Define the system of n-1 equations (Equation 2).

Step 4: Solve for a and b using the method of least squares for n-1 equations

Step 5: Define the tolerance limit (L) allowed by the test method or laboratory

Step 6: Project the uncertainties (Equation 3) until $\hat{U}^{(0)}(k+1) \geq L$

Step 7: Find the period it is needed for, k.

In most cases, effective data modeling is achieved through the use of algorithms, particularly when there is a substantial amount of data available to feed the model. An illustrative example is presented in Figure 4 and Figure 5.

3.2. Weighting of the experimental system based on its traceability chain

If the experimental scheme for the two tests is understood as a single calibration scheme, it is inferred that together, there must be a maximum calibration time for the set.

The idea of an automatic test system (ATS) composed of individual equipment has previously been explored (Jinzhe and Jiulong, 2017a).This idea is a solution to the exaggerated calibration times that result from the application of different techniques for intervals of calibration.

Some equations to determine calibration intervals have in their denominator the difference between the corrections of two successive calibrations. If the correction is equal, the calibration time tends to infinity, which is incorrect from a conceptual point of view. Interpreting the calibration time of the entire test system as a maximum calibration time for any equipment limits these calculation overruns that can appear even in the use of GM(1,1). So that the maximum calibration time can be calculated by Equation 4 for *i* individual equipment:

$$t_{\max} = \sum_{i=1}^{n} t_i w_i \tag{4}$$

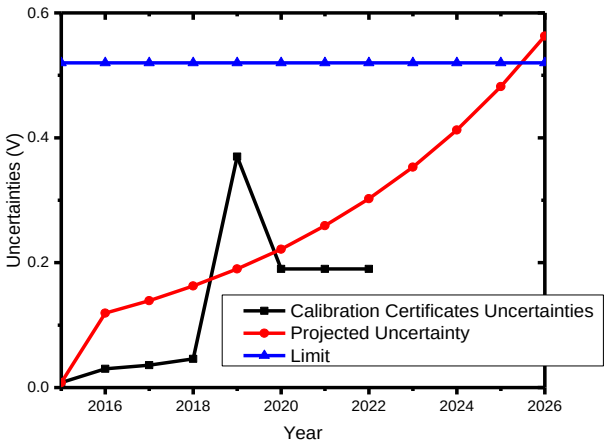

Figure 4 Modeling result by GM(1,1) of the electrical parameter meter (voltmeter) in luminous intensities test in luminaires

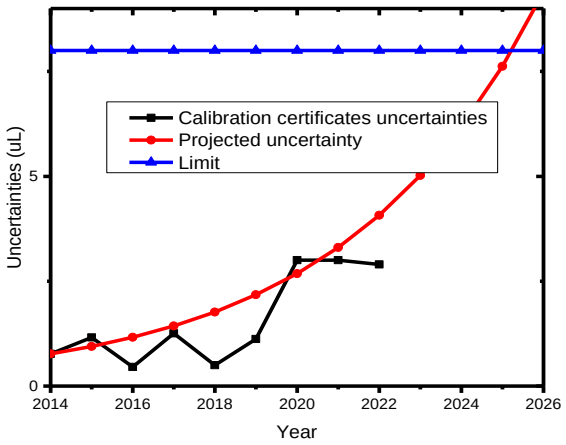

Figure 5 Modeling result by GM(1,1) of a pipette in the gold concentration test

The weighting factor will then be relative to each piece of equipment`s influence on the test result, which can be visualized in the traceability chains. It is possible to create a $Z\zeta$ matrix in which Z represents each piece of equipment and ζ their relationship in the transfer of traceability. If there is a relationship, a value of 1 is associated; otherwise, 0. So, weighting

must follow the relationship in Equation 5 in *j* relations (Jinzhe and Jiulong, 2017b; Wang, Zhang, and Jiang, 2017).

$$w_i = \frac{\sum_j (Z\zeta)_j}{\sum_{ij} (Z\zeta)_{ij}} \tag{5}$$

Results are shown in Table 1. Once the weighting is applied, we get t_{max}=4 years for the Physical-Photometric laboratory and t_{max}=10 years for the Chemical laboratory. Then, the calibration plan must consider the time delivered by the GM(1,1) algorithm for each piece of equipment if it is less than t_{max} and t_{max} if it is greater. Changes in calibration plans represent greater efficiency (Velásquez *et al.*, 2024) in terms of financial resources (Woodhead and Berawi, 2020). The Physical-Photometric and Chemical laboratories pay around 4600 USD and 1000 USD annually, respectively, in a year-to-year calibration plan. The implementation of this methodology has effectively minimized the technical risks associated with the shift calculation, particularly the occurrence of exaggeratedly high values in subsequent calibrations. Additionally, it resulted in savings of 11% and 54% in each case for the following year.

The projection of the behavior of each piece of equipment is obtained by working together as a single experimental scheme in the test, weighing the importance of each one in the traceability chain. This proves to be a critical factor, as obtaining a maximum timeframe within these weight limits not only mitigates the duration of calibration but also enables the formulation of an optimized calibration plan. The methodology tested with chemical and physical laboratories found that the technique can be applied regardless of its particular characteristics.

Table 1 Zζ matrix Physical-Photometric laboratory and Zζ matrix Chemical laboratory

	Chemical laboratory												
	ζ_1	ζ_2	ζ_3	ζ_4	ζ_5	ζ_6	ζ_7	ζ_8	ζ_9	ζ_{10}	ζ_{11}	ζ_{12}	ζ_{13}
Z_1													
Z_2	0	1	1	1	1	1	1	0	0	0	0	0	0
Z_3	0	1	1	1	1	1	1	0	0	0	0	0	0
Z_4	0	1	1	1	1	1	1	0	0	0	0	0	0
Z_5	0	0	1	1	1	1	1	0	0	0	0	0	0
Z_6	0	0	1	1	1	1	1	0	0	0	0	0	0
Z_7	0	0	1	1	1	1	1	0	0	0	0	0	0
Z_8	0	0	1	1	1	1	1	0	1	0	0	0	0
Z_9	0	0	1	1	1	1	1	0	0	0	0	0	0
Z_{10}													
Z_{11}													
Z_{12}													
Z_{13}													
Z_{14}	0	0	1	1	1	1	1	0	1	0	0	0	0
Z_{15}													
Z_{16}	0	0	0	0	0	1	0	0	0	0	1	1	1
Z_{17}	0	0	0	0	0	1	0	0	0	0	0	1	1
Z_{18}	0	0	1	1	1	1	1	0	0	0	0	0	0
Z_{19}	0	0	1	1	1	1	1	0	0	0	0	0	0
Z_{20}	0	0	1	1	1	1	1	0	0	0	0	0	0
Z_{21}	0	0	1	1	1	1	1	0	0	0	0	0	0
Z_{22}													

	Physical-Photometric laboratory							
	ζ_1	ζ_2	ζ_3	ζ_4	ζ_5	ζ_6	ζ_7	ζ_8
Z_1								
Z_2	0	1	1	0	0	0	0	0
Z_3	0	1	1	0	0	0	0	0
Z_4								
Z_5	0	0	0	0	1	0	0	0
Z_6	0	0	0	0	1	0	0	0
Z_7								
Z_8								
Z_9	0	0	0	0	0	0	0	1
Z_{10}								

4. Conclusions

The ISO/IEC 17025 standard requires the calibration of systems and equipment. Calibration intervals must be flexible, but the options for calculating them can generate some technical complications. The main problem is a long calibration interval in which the technical concept of "calibration" loses meaning. The gray model GM(1,1) applied to the uncertainty of calibration of historical data allows for the projection of the uncertainty of equipment for the following period. The tolerance defined by the laboratory or the test/calibration method is a limit with which we can compare the projected values and satisfactorily estimate the next calibration period. The GM(1,1) technique has considerable technical efficiency and saves the economic resources allocated to calibration in the year-to-year plan. Two different tests were studied with this methodology. The results are applicable in the physical or chemical nature of laboratories. There was a reduction of 11% and 54% in financial resources, respectively, while maintaining technical confidence. However, if this methodology is applied, it is essential to have a verification plan to maintain the security of its technical competence with additional measures. Based on the results obtained, interesting future work is to model a risk analysis associated with the differential model used.

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