UDC [633.1+633.85]:001.891.53:[681.2+681.5]:519.876.5
DOI: 10.15673/swonaft.v88i2.3044

THE USE OF MATHEMATICAL MODELLING TO ESTABLISH THE INTERCALIBRATION INTERVAL OF MEASURING LABORATORY INSTRUMENTS OF GRAIN LABORATORIES

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Abstract. *Calibration of measuring instruments is a critical aspect of ensuring the accuracy, reliability, and standardization of quality and safety assessments in grain laboratories. Precise measurements are essential for evaluating key agricultural product parameters, including moisture content, protein concentration, and contaminant levels, which directly influence market value, food safety, and regulatory compliance. Regular calibration of laboratory equipment is mandated by ISO/IEC 17025:2017, which establishes general requirements for the competence of testing and calibration laboratories. This standard ensures measurement traceability, harmonization with international benchmarks, and reproducibility of analytical results. However, frequent calibration procedures present a significant financial burden for laboratories, particularly those operating under budget constraints. The challenge lies in balancing the need for rigorous quality control with cost-effective calibration management.*

This study explores the optimization of intercalibration intervals by leveraging mathematical modelling, specifically the application of control charts and statistical regression techniques. The research analyzes calibration data from two categories of instruments: simple measuring devices, represented by glass hygrometers (229 calibration certificates), and complex precision equipment, exemplified by automatic pipettes (79 calibration certificates). A comparative assessment of eight mathematical models, including linear, quadratic, cubic, and exponential regression functions, was conducted to determine the most suitable approach for approximating error progression over time. The findings indicate that while linear regression models provide robust and interpretable predictions for simple instruments, nonlinear models, particularly cubic regression, demonstrated superior predictive accuracy for complex equipment, explaining up to 94–96% of the variance in calibration errors.

However, while nonlinear models offer enhanced precision in tracking measurement deviations, they necessitate larger datasets to mitigate the risk of overfitting and erroneous extrapolations. The study underscores the importance of selecting appropriate statistical modelling techniques tailored to the specific characteristics of the instrument being calibrated. By integrating data-driven approaches, laboratories can establish optimal calibration intervals, thereby enhancing measurement reliability while reducing unnecessary calibration costs. The proposed methodology provides a framework for achieving a balance between accuracy, compliance, and economic feasibility in laboratory calibration management.

Key words: calibration intervals; mathematical modeling; grain laboratories; instrument accuracy; nonlinear regression.

Introduction

Calibration of measuring equipment and devices is an important component of the functioning of grain laboratories aimed at determining quality and safety indicators of grain and oil crops in global food supply chain. This procedure is designed to ensure traceability and correctness of measurements, which in turn builds international trust in the results of laboratory activities [1].

The need for regular and systematic calibrations is regulated by such a fundamental normative document as ISO/IEC 17025:2017 and is gaining more and more importance with the strengthening of consumer requirements and the growth of the level of Ukraine's integration into the European Union [2]. However, the calibration of the entire range of measuring instruments, which is necessary to meet the modern needs of laboratory research of agricultural raw materials and products, is an expensive process. These financial costs inevitably affect the cost of laboratory analysis, which emphasizes the need for a well-founded establishment of an intercalibration interval, which will implement a balance between the correctness of the measurement and the economic feasibility of its implementation.

Currently, there are many approaches to determining intercalibration intervals, characterized by varying degrees of complexity and detail. Among the most common approaches, ILAC–G24:2007 defines a method for automatic regulation, control charts and in-service verification. However, none of them is universal and has both advantages and disadvantages.

The most well-founded among these approaches is the method of control charts. This method is based on the placement of data on the dynamics of calibration results on a graph, followed by the calculation of the dispersion and average drift for one intercalibration period - instrumental drift [3]. Possibilities of its use in combination with mathematical modelling tools make it possible to consider statistical data and trends. Thus, forming a conclusion about the need to shorten or lengthen the intercalibration interval based on regression forecasting with an established level of confidence can be an effective tool for solving the stated problem.

The study of the prospects of using a mathematical model as a foundation for forecasting the time of the need for recalibration was studied by both foreign [4, 5] and national scientists [6, 7]. However, all these studies used a simple linear model, which can significantly limit the scope of the approach and lead to unreliable results in the case of nonlinear error dynamics. These risks can have significant consequences within the scope of conducting laboratory research on the quality and safety indicators of grain and oil crops.

The purpose of this study was to study the effect of approximation on the effectiveness of using the control chart method.

Materials and methods

The choice of equipment for evaluating factors that can influence the formation of a mathematical model was formed using risk analysis. The analysis process was implemented using the root cause method, in accordance with IEC 31010:2019. The approach considered such factors as the technical complexity of the equipment, the spread of use in analytical methods, and the contribution to the uncertainty of the measurement results. According to the results of the analysis, the category of "simple" and "complex" equipment is separated. Equipment that is methodologically limited in use, consists of a relatively small number of components, contains a scale that directly measures a certain value is classified as simple.

According to the formed categorization, automatic pipettes were chosen as a sample of the model of "complex" equipment, and glass hygrometers of constant mass were selected as a "simple" one. To conduct the research, statistical data was used, represented by certificates of calibration of the ZVT.

The selection of automatic pipettes is represented by 44 devices that included 11 measuring ranges from 1 to 10 thousand µl. Each measuring range was calibrated at the lowest, middle, and highest level forming a coverage of 12 unique calibration points. In total, the study analysed 229 calibration certificates covering 106 individual calibration points and containing data on 539 errors. The data used was accumulated during the period from 4 to 7 years of operation of the devices.

The selection of hygrometers is represented by 16 devices, which included four measurement ranges of 700–1120, 1180–1300, 1360–1660, 1720–1840 kg/m3. Each measuring range was calibrated over the entire interval in steps of 10 kg/m3, forming a coverage of 100 unique calibration points. In total, the study analysed 79 calibration certificates covering 111 individual calibration points and containing data on 548 errors. The data used were accumulated during a period of 4 to 5 years of operation of the devices.

All calibration results, which were analysed in the research process, underwent a verification procedure considering the uncertainty of calibration, according to the recommendations of ILAC G8:09/2019. In the verification process, the compliance of the calibration results with the requirements established by the device manufacturer according to the device range and a specific calibration point within this range is determined.

The method of estimating the inter-calibration interval was based on the analysis of errors obtained because of calibrations carried out by the accredited service provider LLC "Metrology Service" and SE "Ukrmetrteststandard". The stability of the error over time was analysed from the side of drift and prediction of the probable time of its destabilization, using eight mathematical models [8]: linear, quadratic, cubic, power, ab-exponential, logarithmic, hyperbolic, exponential. Thus, the time, expressed in years, during which a chain of calibrations was carried out with a periodicity of once a year, was established as an independent variable. The error specified in the relevant certificates provided by the accredited calibration service provider was set as an independent variable.

Modelling efficiency was assessed by interpreting coefficient of determination and standard error of regression.

The coefficient of determination was calculated according to the formula (1):

$$R^2 = \left(\frac{n \times \sum x_i y_i - \sum x_i \times \sum y_i}{\sqrt{(n \sum x_i^2 - (\sum x_i)^2) \times (n \sum y_i^2 - (\sum y_i)^2)}} \right)^2 \quad (1)$$

where: R^2 – is the coefficient of determination;
x_i – independent variable;
y_i – dependent variable;
n – is the sample size.

The standard error of the regression was calculated according to the formula (2):

$$A = \frac{1}{2} \times \sum \left| \frac{y_i - \hat{y}_i}{y_i} \right| \times 100\% \quad (2)$$

where: A – is the standard error of the regression;
y_i – is the dependent variable;
$\hat{y}_i$ – is an experimental indicator integrated into the model.

The calculation of the term of the next calibration was determined by solving the equation corresponding to the following formula (3), considering the recommendations of the Department of Physiology and Medicine. n., head department, IPK DUITZ Kotsyuba A.M. [2-4]:

$$Reg(b_0 + b_1 \times x_0) \pm \frac{t_{p,n-2} \times Std_{x_i - y_i}}{\sqrt{n_0 - 2}} \times \sqrt{1 + \frac{n \times (x_0 - n_{0,ave})^2}{\sum (n_{0,i} - n_{0,ave})^2}} = \pm D \quad (3)$$

where: Reg – is a regression model, based on the example of linear regression;
x_0 – is the sought variable;
b_0 – intercept;
b_1 – slope;
t – Student's coefficient at n-2;
Std – standard error;
x_i – individual calibration results at a certain level of measurement;
y_i – is the result of solving the regression equation using xi as x0,
n_0 – the number of calibration certificates taken for analysis;
$n_{0,i}$ – the largest individual serial number of the calibration certificate starting from 0;
$n_{0,ave}$ – is the average value of serial numbers of calibration certificates;
D – is a specification assigned to the result of a measurement made at a certain level.

Results of the study and their discussion

In accordance with the established criteria for evaluating the quality of the mathematical model, two metrics were analysed, including indicators of coefficients of determination and relative standard errors of regression. Given the limited amount of data, the median was chosen as the main measure of central tendency of both metadata and simulation results.

It should also be noted that the dynamics of the analytical data used in this research, in almost half of the cases, did not make it possible to form a power, exponential, and ab-exponential approximation. However, the obtained variants of these models were added to the analytical spectrum.

Descriptive statistics of the coefficients of determination of eight mathematical models of the dynamics of calibration results are presented in Table 1.

Table 1 – Descriptive statistics of coefficients of determination of various models of error dynamics of hygrometers and automatic pipettes

Hygrometers								
Model	A	B	C	D	E	F	G	H
Number of models	111	111	110	55	56	111	110	56
Minimum	0.00	0.02	0.43	0.01	0.00	0.00	0.00	0.00
Maximum	1.00	1.00	1.00	0.90	0.90	0.93	0.97	0.90
Average	0.30	0.55	0.92	0.29	0.27	0.32	0.36	0.27
Median	0.21	0.54	0.96	0.22	0.23	0.23	0.27	0.23
Spread	1.00	0.98	0.57	0.89	0.90	0.93	0.97	0.90
Standard deviation	0.27	0.32	0.10	0.24	0.24	0.28	0.29	0.24
Automatic pipettes								

Model	A	B	C	D	E	F	G	H
Number of models	106	106	106	11	11	106	106	12
Minimum	0.00	0.01	0.01	0.00	0.00	0.00	0.00	0.00
Maximum	0.99	1.00	1.00	0.91	0.69	0.98	0.96	0.69
Average	0.30	0.57	0.81	0.29	0.26	0.34	0.36	0.24
Median	0.23	0.59	0.94	0.20	0.19	0.29	0.32	0.18
Spread	0.99	0.99	0.99	0.91	0.69	0.98	0.96	0.69
Standard deviation	0.29	0.33	0.25	0.30	0.25	0.30	0.31	0.25

A - linear, B - quadratic, C - cubic, D - power, E - ab-exponential, F - logarithmic, G - hyperbolic, H - exponential.

According to the obtained data, the spread of the median coefficients of determination of eight mathematical models used to approximate the results of hygrometer calibration was equal to 0.75. The maximum indicator was 0.96 for the cubic model, and the minimum was 0.21 for the linear model. The cubic model also had the lowest standard deviation of the coefficient of determination, which was 0.10. In general, the value of the standard deviation of the coefficient of determination of the approximation of "simple" equipment was in the range of 0.1-0.32.

The median spread of the coefficient of determination of the approximation of automatic pipette calibration results was 0.76. The maximum indicator was 0.94 for the cubic model, and the minimum was 0.18 for the exponential model. The indicator of the minimum standard deviation of the coefficient of determination was equal to 0.25 and belonged to the cubic model. In general, the value of the standard deviation of the coefficient of determination of the approximation of "complex" equipment was in the range of 0.25–0.33.

The results indicate a high potential for the application of the cubic model, both in the case of "simple" and "complex" equipment. Cubic approximation can explain 94-96% of the variance of the calibration results during the operation of the device. The general distribution of coefficients of determination did not differ significantly between equipment categories.

Descriptive statistics of the relative standard errors of the regression of eight mathematical models of the dynamics of calibration results are presented in Table 2.

Table 2 – Descriptive statistics of relative standard errors of regression (%) of different models of error dynamics of hygrometers and automatic pipettes

Hygrometers								
Model								
Number of models	108	108	107	55	56	108	107	56
Minimum	0.15	0.00	0.00	0.22	0.19	0.22	0.21	0.00
Maximum	726.08	699.45	188.03	49.04	37.16	698.30	653.82	37.16
Average	20.93	18.02	6.14	6.70	6.47	20.10	18.82	6.40
Median	4.93	3.44	1.10	4.14	4.39	4.55	4.21	4.39
Spread	725.93	699.45	188.03	48.82	36.97	698.07	653.61	37.16
Standard deviation	73.79	70.64	20.63	8.58	7.34	70.82	66.47	7.38
Automatic pipettes								
Model								
Number of models								
Minimum	5.50	0.65	0.00	0.00	0.00	5.56	5.41	0.00
Maximum	6363.57	3334.90	1218.19	64.11	82.47	6709.15	6642.14	82.47
Average	198.43	142.34	69.19	28.62	32.91	200.33	198.38	30.17
Median	82.17	60.06	26.49	29.33	30.08	79.22	80.47	27.89
Spread	6358.07	3334.24	1218.19	64.11	82.47	6703.58	6636.72	82.47
Standard deviation	684.66	377.72	158.23	19.92	25.08	717.11	706.08	25.73

A – linear, B – quadratic, C – cubic, D – power, E – ab-exponential, F – logarithmic, G – hyperbolic, H – exponential.

According to the obtained data, the median scatter of the relative regression errors used to approximate the results of hygrometer calibration was equal to 3.83%. The maximum indicator was 4.93% for the linear model, and the minimum was 1.10% for the linear model.

The spread of median relative errors of approximation of automatic pipette calibration results was 55.69%. The maximum indicator was equal to 82.17% in the case of the linear model, and the minimum – 26.49% for the cubic model. The obtained values demonstrate a much lower accuracy of modelling in the case of approximation of "complex" equipment.

Empirical data show the ratio of the median relative error of modelling "simple" and "complex" equipment as 1:17 at the minimum and 1:24 at the maximum levels. This demonstrates a rather small difference in reliability between all analysed mathematical models with respect to "simple" equipment. At the same time, the reliability of modelling by all approximation variants significantly deteriorates in terms of "complexity". This conclusion is confirmed by the standard deviation of modelling errors, which took the values of 7.34-73.79 for approximations of "simple" equipment, and 19.92-717.11 for "complex" ones.

For both categories of equipment, the least reliable modelling option was found to be the linear model, which had the lowest determination index and the lowest error index. The greatest prospects of use are demonstrated for the use of the cubic model.

To evaluate the influence of the model on the calculation of the intercalibration interval, using the established method, an analysis of the results of the integration of the obtained linear and cubic regression equations was carried out.

To identify the possible occurrence of erroneous data, calculations were performed at two levels of significance, which included $p=0.95$ and $p=0.99$. The results of the solution were evaluated in three ways.

– the model was considered workable if there was a solution $p=0.95 > p=0.99$;
– the model was considered incorrect in the case of $p=0.95 < p=0.99$;
– the model was considered unworkable in the absence of a solution.

The results of the analysis are presented in Table 3.

Table 3 – Results of the assessment of the influence of the use of linear and cubic mathematical models on the calculation of intercalibration intervals

Hygrometers	Linear model		Cubic model	
	Quantity	Percent	Quantity	Percent
Working model	104	93.69	35	31.53
Incorrect model	4	3.6	74	66.67
Not working model	3	2.7	2	1.8
Total number	111		111	
Automatic pipettes	Linear model		Cubic model	
	Quantity	Percent	Quantity	Percent
Working model	62	58.49	60	56.6
Incorrect model	7	6.6	44	1.51
Not working model	37	34.91	2	1.89
Total number	106		106	

According to the obtained data, despite the higher quality statistical data that highlighted the potential of using the cubic model, for "simple" equipment, the use of the linear model was more effective. Replacing linear regression with cubic led to a threefold decrease in predictability with the formation of a significant number of false results. Considering the equivalence of the number of unworkable equations, which ranged from 1.8 to 2.7%, this effect is most likely caused by the lack of data for effective training of the cubic model. However, given the difference between the standard error of linear and cubic regression, which were 4.93% and 1.10%, respectively, the linear model can be used to reasonably calculate the intercalibration interval of "simple" equipment.

Replacing linear regression with cubic led to an almost two-fold increase in performance, which, however, was accompanied by a six-fold increase in the number of false positives, which is also likely due to the limitation of the analytical sample. However, given the significant difference between the regression standard errors in complex equipment, which was 82.17% and 26.49% for linear and cubic regression, respectively, the use of the linear model in the case of "complex" equipment is significantly limited.

Given the industry risks inherent in the food and feed industry, we do not consider it appropriate to use a linear model to calculate the inter-calibration intervals of critically important "complex" equipment, which may include such categories of equipment as automatic pipettes, scales, and thermal equipment.

Conclusions. The application of mathematical models for calculating the interval between the calibration of measuring equipment is an important direction in the field of ensuring the reliability of the results of laboratory research on the quality and safety indicators of grain and oil crops.

Linear regression proves to be the most successful option for modelling the error dynamics of "simple" equipment, despite the relatively low coefficient of determination and relatively high values of standard errors.

This type of regression, however, may not be applicable to "complex" equipment with high variance and erratic trends in calibration results.

Within the framework of the conducted research, the cubic model was found to be the most effective approximation method for "complex" equipment. Nevertheless, when estimating the intercalibration interval for this type of equipment, it is necessary to conduct a preliminary analysis to determine the optimal form of approximation.

It should be considered that nonlinear approximations may be inefficient or unsuitable for extremely limited input data, which is often encountered in practice. However, with proper accounting of risk factors, the cubic model can be a reliable method of estimating the intercalibration interval.

Thus, the application of mathematical models for calculating the interval between calibrations is promising but requires a careful analysis of the characteristics of the measuring equipment and the selection of an adequate type of approximation. It is also important to consider the limitations associated with the amount of available data to increase the reliability of the results.

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ВИКОРИСТАННЯ МАТЕМАТИЧНОГО МОДЕЛЮВАННЯ ДЛЯ ВСТАНОВЛЕННЯ МІЖКАЛІБРУВАЛЬНОГО ІНТЕРВАЛУ ЗАСОБІВ ВИМІРЮВАЛЬНОЇ ТЕХНІКИ, ЩО ВИКОРИСТОВУЄТЬСЯ ДЛЯ АНАЛІЗУ ПОКАЗНИКІВ ЯКОСТІ ТА БЕЗПЕЧНОСТІ ЗЕРНОВИХ ТА ОЛІЙНИХ КУЛЬТУР

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Анотація. *Калібрування вимірювальних приладів є критично важливим аспектом забезпечення точності, надійності та стандартизації оцінки якості та безпеки в зернових лабораторіях. Точні вимірювання необхідні для оцінки ключових параметрів сільськогосподарської продукції, включаючи вміст вологи, концентрацію білка та рівні забруднювачів, які безпосередньо впливають на ринкову вартість, безпечність харчових продуктів і відповідність нормативним вимогам. Регулярне калібрування лабораторного обладнання передбачено стандартом ISO/IEC 17025:2017, який встановлює загальні вимоги до компетентності випробувальних і калібрувальних лабораторій. Цей стандарт забезпечує простежуваність вимірювань, гармонізацію з міжнародними стандартами та відтворюваність аналітичних результатів. Однак часті процедури калібрування становлять значний фінансовий тягар для лабораторій, особливо тих, які працюють в умовах бюджетних обмежень. Проблема полягає в тому, щоб збалансувати потребу в суворому контролі якості з економічно ефективним управлінням калібруванням.*

У цьому дослідженні досліджується оптимізація міжкалібраційних інтервалів шляхом використання математичного моделювання, зокрема застосування контрольних карт і методів статистичної регресії. У дослідженні аналізуються дані калібрування двох категорій інструментів: простих вимірювальних пристроїв, представлених скляними гігрометрами (229 сертифікатів калібрування), і складного прецизійного обладнання, прикладом яких є автоматичні піпетки (79 сертифікатів калібрування). Порівняльна оцінка восьми математичних моделей, включаючи лінійну, квадратичну, кубічну та експоненціальну регресійні функції, була проведена для визначення найбільш прийнятного підходу для апроксимації прогресії помилки з часом. Отримані результати показують, що в той час як моделі лінійної регресії забезпечують надійні та інтерпретовані прогнози для простих приладів, нелінійні моделі, зокрема кубічна регресія, продемонстрували високу точність прогнозування для складного обладнання, пояснюючи до 94–96% відхилень у помилках калібрування.

Однак, незважаючи на те, що нелінійні моделі пропонують підвищену точність у відстеженні відхилень вимірювань, вони вимагають більших наборів даних, щоб зменшити ризик переобладнання та помилкових екстраполяцій. Дослідження підкреслює важливість вибору відповідних методів статистичного моделювання, адаптованих до конкретних характеристик приладу, що калібрується. Завдяки інтеграції підходів, керованих даними, лабораторії можуть встановлювати оптимальні інтервали калібрування, тим самим підвищуючи надійність вимірювань і одночасно знижуючи непотрібні витрати на калібрування. Запропонована методологія забезпечує основу для досягнення балансу між точністю, відповідністю та економічною доцільністю в управлінні лабораторним калібруванням.

Ключові слова: міжкалібрувальні інтервали; математичне моделювання; зернові лабораторії; точність приладу; нелінійна регресія.

Отримано в редакцію 05.09.2024 Received 05.09.2024
Прийнято до друку 19.09.2024 Approved 19.09.2024