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Research Article

Comparative Analysis of the Various Statistical Techniques Applied to Assess Participants' Laboratory Performance in an Ordinary Portland Cement (OPC) Proficiency Testing Program

Shwet Vashishtha*

R. P. P. T. Laboratory, National Test House (NR), Ghaziabad, India

Abstract

Outliers in data analysis pose both challenges and opportunities for researchers. On one hand, if not adequately addressed, outliers can distort statistical analyses and lead to flawed conclusions. Conversely, outliers can also offer valuable insights into underlying processes or factors at play. One commonly used method for identifying outliers is through the analysis of interquartile ranges (IQRs). By accurately detecting and treating these anomalies, researchers can ensure the accuracy and validity of their findings. The major causes of outliers in data analysis stem from measurement and sampling errors. These errors can arise from issues such as human errors in data collection or problems with measurement equipment. Researchers must comprehend these causes to appropriately address outliers and minimize their impact on the analysis. Treating outliers effectively can greatly enhance data analysis by providing a more precise representation of underlying patterns and relationships. Removal or adjustment of extreme values enables researchers to obtain a clearer and more reliable picture of the phenomena under investigation, leading to crucial insights and facilitating further analyses and decision-making. Addressing outliers also offers opportunities for additional research and a deeper understanding of the underlying processes or factors at play. By extensively investigating the reasons behind outliers, researchers can gain valuable insights that can guide future research efforts and contribute to more informed decision-making based on the data. An exemplary illustration of the significance of accurate assessment techniques in statistical analyses is the OPC fineness study. This study analyzed the impact of various assessment methods on scoring results by comparing data from different laboratories using z-scores. The findings of this study demonstrated that the choice of assessment technique significantly influenced the scoring outcomes. Therefore, careful consideration of assessment procedures is crucial for obtaining reliable and comparable results in statistical analyses. In conclusion, outliers in data analysis present both challenges and opportunities for researchers. Accurately detecting and addressing outliers is essential for obtaining reliable and meaningful results. A comprehensive understanding of the causes of outliers, such as measurement and sampling errors, is necessary for appropriate treatment. Effectively treating outliers enhances the accuracy and validity of analysis and provides avenues for further research and informed decision-making. The OPC fineness study exemplifies the importance of assessment techniques in statistical analyses. A nuanced understanding of outlier detection and treatment is indispensable for drawing valid statistical conclusions.

Keywords

Proficiency Testing, z-score, Statistical Techniques, Outliers, OPC

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*Corresponding author: shwetraj@gmail.com (Shwet Vashishtha)

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1. Introduction

Programs for proficiency testing are intended to assess participating laboratories' analytical capabilities. They make it possible to evaluate experimental tests that are regularly carried out critically, spot analytical issues, and make it easier to put the required corrective measures into place. The provider of the proficiency testing (PT) program is in charge of carrying out the statistical analysis and offering a performance indicator for each participant.

The most important international standards for measurement control in laboratories are the ISO 5725 standards with the group name "Accuracy (correctness and precision) of measurement methods and results" (6 parts) [1–6]. The international standard ISO/TR 22971 [7] provides users with practical guidance on the application of ISO 5725-2 [2] and presents simplified step-by-step procedures for the design, implementation and statistical analysis of interlaboratory testing to assess variability Standard measurement method and for determining the repeatability and reproducibility of data from interlaboratory tests. The ISO/IEC 17043 [8] standard specifies general requirements for the competence of proficiency testing program providers and for the development and operation of proficiency testing programs. These requirements are intended to apply generally to all types of proficiency testing programs and may be used as a basis for specific technical requirements for particular areas of application. The international standard ISO 13528 [9] contains detailed descriptions of statistical methods for data analysis, verified against qualification tables.

The main objective in organizing these proficiency tests is to assess the technical competence of the laboratory to carry out measurements and also to meet the requirements of ILAC/APLAC regarding the compatibility of the results submitted by these laboratories. Participation in the proficiency testing program/laboratory comparison is mandatory for National Accreditation Board for Testing and Calibration Laboratories (NABL) accredited cement testing laboratories as per ISO/IEC 17025:2017 [10]. The laboratories carried out suitability tests for physical test parameters, i.e. the density [11] of ordinary Portland cement. First, the Z-scores for the results of the participating laboratories are evaluated based on the median and NIQR. The NIQR is determined as 0.714 x IQR (IQR is the difference between the 3rd and 1st quartiles). The Z-scores demonstrate the laboratory's ability to competently perform the above analyses. Proficiency testing programs are statistical quality assurance programs that allow laboratories to evaluate their performance in conducting test methods in their own laboratories by comparing their data with those of other laboratories participating in the same program. The aim of the aptitude test is to independently assess the competence of the participants.

Evaluating various statistical methods is important to evaluate the performance of each laboratory in PT programs. It is certainly important for providers and participants of public service programs to know whether there would be significant differences in the assessment results when using different methods of performance statistics. Based on these considerations, this study compared the suitability of different statistical approaches for determining the allocation value and standard deviation for performance evaluation. This work also aimed to improve the statistical approach currently used by this proficiency testing program. The results obtained were evaluated using different statistical approaches, viz. Cochran test, Grubb test, Hampel test, Dixon test, Mandel method, nIQR method, robust algorithm – a statistical method and classic Z-score to provide the best method to represent the participant's performance along with outlier detection to determine. When the number of data is larger and there are large differences between laboratory results, detecting outliers is a difficult task in this case. Therefore, in the present work, emphasis was placed on developing a suitable method for determining the outlier so that the extreme values reported by some PT participants do not influence the results of participants close to the reference value.

The Z-score is the performance score of a laboratory compared to the other laboratories. The quality of the measurement increases as the Z-score value decreases. In this study, laboratories with a Z-score value between ±2 and ±3 are considered questionable. It is recommended to examine your results carefully to resolve the error. The limit value of the Z-score evaluated by any method is outside the range of ±3, indicating that there is a problem in the measurement.

2. Statistical Techniques

The statistical analysis of the data obtained from the participants was carried out in this article using both numerical techniques such as Cochran, Grubb, Hampel and Dixon statistics and graphical techniques such as the classic Z-score, Median & NIQR, Robust Algorithm-A and statistics illustrates method [12-13].

2.1. Grubb's Method

Grubbsused to detect a single outlier in a univariate data set. The data set that follows an approximately normal distribution. Grubbs' test is defined as the following two hypotheses:

H0: There is no outlier in the data set

H1: There is at least single outlier in the data set

The general formula for Grubbs' test statistic is defined as:

$$G = \max \left| yi - y \right| / s$$

Where y_i is the element of the data set, y and s denoting the sample mean and standard deviation and the test statistic is the largest absolute deviation from the sample mean in units of the sample standard deviation. The calculated value of parameter G is compared with the critical value for Grubb's test. When the calculated value higher or lower than the critical value of choosing statistical significance, then the calculated value can be accepted as and outlier. The statistical significance (α) describes the maximum mistake level which a person searching for outlier can accept.

2.2. Dixon's Method

The test developed by Dixonand used to the test is appropriate for small sample size. The test has some limitations to n≤30, were later on extended to n≤40. The test first step for organizing the data in an ascending order, and then the next step is to count parameter R.

The test has various test statistics. Suppose for testing large set of elements to be an outlier, the sample arranged in ascending order X1≤X2≤.… ≤Xn implying that the large sample element is given by Xn. Dixon proposed the following test statistics defined as

$$R_{10}=x_n-x_{n-1}/x_n-x_1,\ 3\le n\le 7$$

$$R_{11}=x_n-x_{n-1}/x_n-x_2,\ 8\le n\le 10$$

$$R_{21}=x_n-x_{n-2}/x_n-x_2,\ 11\le n\le 13$$

$$R_{22}=x_n-x_{n-2}/x_n-x_3,\ 14\le n\le 30$$

For testing the smallest sample element to be an outlier, the sample is ordered in descending order implying that the smallest sample element is labeled Xn. All the selection of the test statistics depends on the Dixon's criteria.

The variable $X_n$is marked as an outlier, when the corresponding statistic (n) exceeds a critical value, which depends on the selected significance level α.

The calculated value of the parameter R is compared with the Dixon's test critical value for choosing statistical significance. When the calculated value of parameter R is bigger than the critical value then it is possible to accept data from the data set as an outlier.

2.3. Hampel's Method

To calculate Hampel's test statistical tables are not necessary. Theoretically, this method is resistant, which means that it is not sensitive to outliers, it also has no restrictions as to the abundance of the data set. Hampel's test performs the steps for data sets are as follows:

i. Compute the median (Me) for the total data set. The median is described as the numeric value and separating the higher half of a data set from the lower half.

ii. Compute the value of the deviation $ri$from the median value; this calculation should be done for all elements from the data set:

$$r_i=(x_i-\text{Me})$$

where, $x-$ simple data from the data set, $i-$ belongs to the set for 1 to n, $n-$ number of all element of the set and $Me-$ median

iii. Calculate the median for deviation $Me_{|ri|}$

iv. Check the conditions: $|r_i|\ge 4.5Me_{|ri|}$

If the condition is executed, then the value from the data set can be accepted as an outlier.

2.4. Cochran's Method

According to ISO 5725-2, Cochran's test is recommended for the detection of outliers in a given set of intra laboratory variability test. It is a one – sided outlier test as the criterion of the test examines only the greatest standard deviation and allows to eliminate the problematic result with the within laboratories reproducibility / repeatability. Cochran's statistic C is calculated using the following formula

$$C = SD^2_{max}/\Sigma^{p}_{j=1}SD_j^2$$

where, SD_{max} is the maximum standard deviation among the investigated laboratories

SD_j is the standard deviation of data from the laboratory

p is the number of participated laboratories

The calculated C value can be compared with the critical value for a given n value i.e. the number of results given by each laboratory.

2.5. Mandel's k/h Method

Mandel's k and h consistency test statistics are discussed in ASTM E691 [14] standards for inter-laboratory analysis. 'k' value is a measure of within-laboratory consistency in repeatability and the 'h' test statistic is used to examine the consistency of interlaboratory data, confirming if any laboratory data is an outlier. In other words, it is to indicate the accuracy of a lab results against the others reported. 'h' test statistic value reflects the deviation of a single laboratory's mean test results from the overall mean results obtained from all participating laboratories. 'h' and h_{crit} are a measure of seriousness in a lab's inaccuracy and define as;

$$h_j = d_j / S_x$$

Where, d_j is deviation of mean result of a lab (j) from the overall mean and S_x is the standard deviation

$$h_{crit} = t\,(p-1)/\sqrt{\{p(t^2 + (p-2)\}}$$

Where, t is the student's distribution with degree of freedom v=p-2 and α = 0.05

p is the number of participating laboratories

When the 'h' value is larger than the h_{crit} it is concluded that the mean result given by the laboratory concerned is not accurate and reliable.

2.6. Normalized Interquartile Range (nIQR) Method

The process of the performance evaluation of the participating laboratories is illustrated by the z-score values of the results obtained in the PT program in accordance to the ISO 13528 guidelines. The values of standardized sum (S_i) and standardized difference (D_i) between two results of laboratory "i" have been calculated using equations.

$$S_i = (A_i + B_i) / \sqrt{2}$$

$$D_i = (A_i - B_i) / \sqrt{2} \text{ if median } (A_i) > \text{median } (B_i)$$

Whereas, A_i and B_i are the two measurement values of the laboratory 'i'

Using the values of S_i and D_i the values of both the Z scores i.e Z-score between the laboratories (Zbi) and within the laboratory (Zwi) can be calculated using equations.

Zbi = Si – Median (Si)/IQR (Si) x 0.7413 (Variation between the laboratory: Reproducibility)

Zwi = Di – Median (Di)/IQR (Di) x 0.7413 (Variation within the laboratory: Repeatability)

The interquartile range (IQR) is the difference between the lower and upper quartiles of data. The lower quartile (Q1) is the value below, which a quarter of results laid and the upper quartile (Q3) is the value above, which a quarter of results laid. The quartiles are calculated analogously to the median and IQR = Q3 - Q1. The term "Normalized IQR" is comparable to a standard deviation and equals to IQR x 0.7413. The factor 0.7413 comes from the standard normal distribution, which has a mean of zero and a standard deviation equal to one. The width of the interquartile range of such distribution is 1.34898 and 1/1.34898 = 0.7413. z-score values Zbi and Zwi of laboratory "i" are the robust z-score of its *i S* and *i D* values. Finally, each participating laboratory will be assigned with two z-score values on the basis of their results. Generally, laboratories having the value of any z-score value outside the range ± 3 are considered to be the outlier laboratories.

2.7. Robust Algorithm-A Method

The robust statistics can be calculated according to ISO 13528: Robust analysis Algorithm A. The robust average (x*) and the standard deviation value (s*) of the results of the participants could achieve by an iterative calculation as described in ISO 13528 and is not affected by the results far from the reference value. Performance is evaluated by calculating z-score or z′-score (z prime) in the given expression as the uncertainty of the assigned value $u(x_{pt}) < 0.3\sigma_{pt}$. The Z′ score is calculated as follows;

$$Z' = (x_i - x_{pt}) / \sqrt{\sigma^2_{pt} + u^2(x_{pt})}$$

where, x_i is the test result from participant laboratory, x_{pt} is the assigned value and σ_{pt} is the standard deviation for proficiency assessment (SDPA).

$$u(x_{pt}) = 1.25s^*/\sqrt{p}$$

where, $u(x_{pt})$ is the uncertainty of assigned values, s* is robust standard deviation and p is number of participants

2.8. Classical Z-score Method

The z-score is the score given to the participant as per their performance. The classical z-score can be calculated as follows

$$z = (X_{lab} - X_{mean})/SD$$

where, X_{lab} is the result of the individual laboratory X_{mean} is the mean value of the analyte obtained by the participants result and SD is the standard deviation of the data.

3. Sample Details of PT Items and Methodology

The proficiency testing program aimed to assess the accuracy and precision of laboratories in determining the density of Regular Portland Cement Grade 53. The selected statistical methods, including Grubb's, Dixon's, Hampel's, Mandel's, Cochran's, nIQR, Robust Algorithm-A, and Classical Z-Score, were systematically applied to the dataset. These methods were chosen for their ability to detect outliers and provide a robust evaluation of laboratory performance.

Laboratories received ISI-marked cement samples conforming to IS 269:2015 standards. The samples underwent thorough dry mixing, coning, and quartering following IS 3535-1986 to ensure homogeneity and representativeness. Density determination was conducted using the IS 4031 (Part 11): 1988 standard.

The results obtained from the 13 participating laboratories were recorded as Table 1 and analysed using the selected statistical techniques. Z-scores were calculated, and outliers were identified based on predefined criteria. The consistency of outlier detection across methods and the impact on cross-laboratory comparisons were assessed.

Table 1. *Details of Laboratory Results.*

S.NO.	Lab Code	Density (g/cc)	
		Result-A	Result-B
1	A	3.15	3.11
2	B	3.15	3.15
3	C	3.14	3.15
4	D	3.14	3.14
5	E	3.14	3.14
6	F	3.15	3.14
7	G	3.14	3.15
8	H	3.17	3.12
9	I	3.20	3.21
10	J	3.13	3.14
11	K	3.07	3.10
12	L	3.17	3.17
13	M	3.12	3.14

4. Result and Discussion of Statistical Analysis

In proficiency testing programs, the results obtained by participating laboratories are usually expressed in the form of a Z-score to represent their performance. The individual laboratory's Z-score indicates how much the reported result deviates from the reference/assigned value and which results are classified as satisfactory, questionable and unsatisfactory. In this laboratory performance evaluation work, the data obtained from the participating laboratories were converted into the Z-score using statistical methods. According to the classic Z-score, the outlier results affect the mean and standard deviation of the data set. In the classical approach, the maximum number of laboratories shows that the Z-score is within an acceptable range, i.e. H. within the Z-score ± 2. The Z-score determined using the classic, robust and nIQR method is shown in Table 3. In ISO 13528 Robust analysis of the algorithm-A method to obtain robust estimates. First, the PT results were arranged in ascending order and the absolute deviation from the median was calculated. Then the process converges through iteration, i.e. by updating the robust average (x*) and robust standard deviation (s*) several times so that the value does not change from one iteration to the next iteration up to the third digit. The robust average is considered as the assigned value and the uncertainty value was derived by substituting the robust standard deviation into the equation. The Z-score was calculated using the robust estimates. The laboratories identified as outliers using this method are similar to the laboratories found in the case of the Grubb, Dixson, Mandel and classical methods for density test parameters. From the analysis of the data, it appears that this is the best method to derive the assigned value when the reference value for PT is not known.

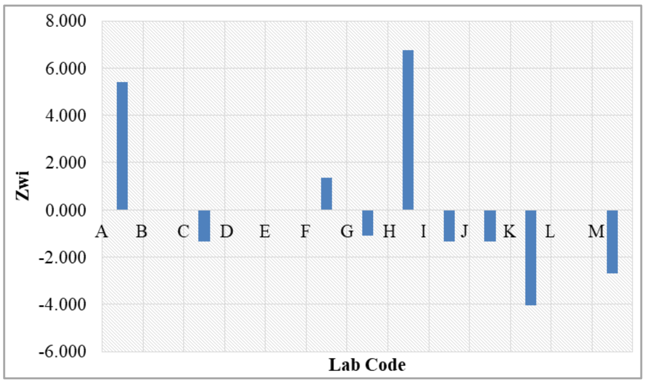
Table 2. *Outliers detected by Various Statistical Approaches.*

Statistical Methods	Density
Grubb's Method	I and K
Dixson's Method	I and K
Hampel's Method	I, K and L
Cochran's Method	Nil
Mandel's Method	I and K
nIQR Method	
Zbi	I, K and L
Zwi	A, H, K and M
Robust Algorithm-A Method	I and K
Classical Method	I and K

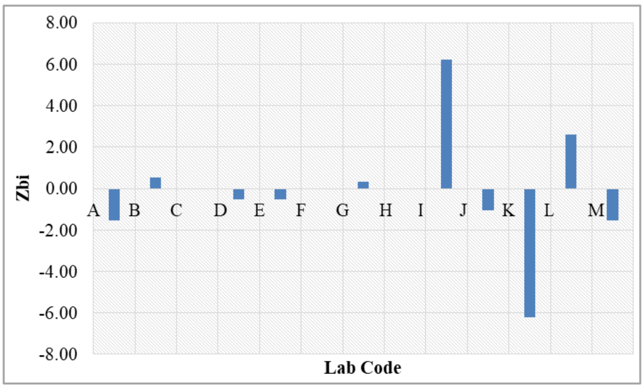
Table 3. *Z-score obtained by classical, robust and niqr methods.*

Lab Code	Value (g/cc)	z-score classical	z-score Robust	z-score by nIQR (Zbi)
	3.13	-0.516	*-0.889*	-1.56
	3.15	0.238	*0.498*	0.52
	3.15	0.049	*0.151*	0.00
	3.14	-0.139	*-0.196*	-0.52
	3.14	-0.139	*-0.196*	-0.52
	3.15	0.049	*0.151*	0.00
	3.15	0.162	*0.359*	0.31
	3.15	0.049	*0.151*	0.00
	3.21	2.312	*4.314*	6.23
	3.14	-0.328	*-0.543*	-1.04
	3.09	-2.214	*-4.011*	-6.23
	3.17	0.992	*1.886*	2.59
	3.13	-0.516	*-0.889*	-1.56

The data received from the participating laboratories were also quantitatively evaluated for their measurement quality using the nIQR method. For this purpose, the PT data was processed to calculate two types of Z-scores such as the Z-score within the laboratory (Zwi) and between the laboratories (Zbi). An intra-laboratory z-score value indicates the variance in data generated in the same laboratory, and an inter-laboratory z-score indicates the variance in data generated by different participating laboratories. The quality of the measurement increases as the Z-score value decreases. The values of both the intra-laboratory Z-score and the inter-laboratory Z-score were calculated for the parameters and plotted against the laboratory code (Figure 1a and 1b). Using the Z-score results chart, each laboratory can easily compare its performance with its own results and those of other laboratories.



(a)



(b)

Figure 1. *(a) Z-score within the laboratory (Zwi) obtained by nIQR Method. (b) Z-score between the laboratory (Zbi) obtained by nIQR Method.*

The consistent identification of Laboratories I and K as outliers across various statistical methods raises questions about the accuracy and precision of their density determination. These findings emphasize the importance of investigating the root causes of discrepancies in these laboratories. Possible factors contributing to outliers could include instrument calibration issues, methodological deviations, or inadequate training.

The absence of outliers identified by Cochran's method suggests a relative homogeneity in the majority of laboratories, indicating a consistent approach to density determination. This aligns with the purpose of proficiency testing programs to ensure the compatibility of results across different laboratories.

The additional outlier identified by Hampel's and nIQR methods in Lab L adds a layer of complexity to the analysis. The utilization of multiple statistical techniques is crucial for a comprehensive evaluation of laboratory performance. Lab L's outlier status implies a potential issue that might not have been detected by other methods.

The Z-score analysis further confirms the significant deviations in Labs I, K, and L. These laboratories exhibit Z-scores well beyond the acceptable range, indicating a need for corrective actions to improve the accuracy and reliability of their density determinations.

The comparative analysis of various statistical techniques applied to assess participants' laboratory performance in an Ordinary Portland Cement (OPC) proficiency testing program for density determination has provided valuable insights into the reliability and accuracy of the results. The utilization of statistical methods, including Grubb's, Dixon's, Mandel's, nIQR, Robust, and Classical approaches, consistently identified outliers, with Laboratories I and K standing out as consistent outliers across multiple methods. The Z-score analysis further confirmed the substantial deviations exhibited by these laboratories, emphasizing the need for targeted investigations to address procedural issues or other factors impacting their accuracy.

Laboratory performance evaluation, guided by statistical techniques, plays a crucial role in ensuring the quality of density determination results for regular Portland cement grade 53. The observed consistency in outlier detection across various methods underscores the reliability of the findings and highlights the significance of employing multiple statistical approaches for a comprehensive assessment.

Furthermore, the absence of outliers detected by Cochran's method suggests a general homogeneity in the data from the majority of participating laboratories, reflecting a commendable consistency in density determination methodologies. This collective reliability is crucial for the proficiency testing program's overall effectiveness and underscores the laboratories' competence in generating accurate and comparable results.

The recommendations for continuous improvement, including targeted investigations into the procedures of outlier laboratories and the consideration of robust statistical methods, serve as actionable steps to enhance laboratory practices. The comprehensive analysis presented in this paper contributes to the ongoing efforts to refine proficiency testing programs, ensuring the quality and reliability of density determinations in the field of cement testing.

In summary, this research underscores the importance of employing a diverse set of statistical methods for proficiency testing assessments, providing laboratories with valuable insights for continuous improvement and contributing to the overall quality assurance in the analysis of Ordinary Portland Cement.

5. Conclusions

In conclusion, the comparative analysis of various statistical techniques applied to assess participants' laboratory performance in an Ordinary Portland Cement (OPC) proficiency testing program for density determination has provided valuable insights into the reliability and accuracy of the results. The utilization of statistical methods, including Grubb's, Dixon's, Mandel's, nIQR, Robust, and Classical approaches, consistently identified outliers, with Laboratories I and K standing out as consistent outliers across multiple methods. The Z-score analysis further confirmed the substantial deviations exhibited by these laboratories, emphasizing the need for targeted investigations to address procedural issues or other factors impacting their accuracy.

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In summary, this research underscores the importance of employing a diverse set of statistical methods for proficiency testing assessments, providing laboratories with valuable insights for continuous improvement and contributing to the overall quality.

Abbreviations

IQR: Interquartile Range
NTH: National Test House
OPC: Ordinary Portland Cement
PT: Proficiency Testing
PTP: Proficiency Testing Program
NABL: National Accreditation Board for Testing and Calibration Laboratories

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Conflicts of Interest

The authors declare no conflicts of interest.

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