Robust Statistics Analysis of Proficiency Test Result of Cement

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Abstract

A proficiency testing scheme for cement testing according to IS specifications was set up for cement testing laboratories in India. Proficiency testing schemes are inter-laboratory performance comparisons allowing participants to evaluate themselves against pre-established criteria. Proficiency testing is a powerful tool to help laboratories improve their performance as well as demonstrate their competences to accreditation bodies or customers. 24 cement testing laboratories participated in this program. Proficiency test results of physical test parameters of Ordinary Portland Cement-53 grade were obtained through organizing and performing the cement proficiency test plan. After performing the robust statistical analysis of the results, the test proficiency of the laboratory is evaluated.

Keywords: Cement, Proficiency Testing, Robust "z-score"

I. INTRODUCTION

The primary objective for organizing this proficiency testing (PT) is to assess the laboratory's technical competence to perform measurements and also fulfilling the requirement of ILAC/APLAC with regard to the compatibility of results submitted by these laboratories. Participation in Proficiency Testing Programme (PTP)/Inter Laboratory Comparison (ILC) is mandatory for NABL accredited cement testing laboratory. The laboratory carries out three physical test parameters namely, Soundness by Le-Chatelier Method, Standard Consistency, and 7 days Compressive Strength according to IS 4031 (part 3, 4 & 6):1988. The results are evaluated by the robust analysis Algorithm 'A' method as per Clause 9.4 of ISO 13528:2015 by calculating the Robust Z Score of the test parameter of each participant laboratory. The z-scores demonstrating the laboratory's ability to competently perform the above mentioned analyses. Proficiency testing programs are statistical quality assurance programs that enable laboratories to assess their performance in conducting test methods within their own laboratories when their data are compared against other laboratories that participate in the same program. The aim of Proficiency testing is to provide an independent assessment of the competence of participating laboratories. Together with the use of validated methods, Proficiency testing is an essential element of laboratory quality assurance. The PT programs conducted by NTH (WR) are independent schemes arranged by an independent section in PTP Division. The PT designed for laboratories to ensure the performance of individual laboratories for specific tests or measurements, and to monitor the continuing performance of laboratories. It provides laboratories with an objective means of assessing the reliability and confidence in the data they are producing. It also complies with the requirements of ISO/IEC 17025:2017: 7.7 – Ensuring the validity of results – clause 7.7.2 (a) "participation in proficiency testing".

The PTP division of National Test House (WR) has been accredited by NABL as PT provider in accordance with ISO/IEC 17043:2010 for operating the simultaneous PT schemes in Cement, Flyash and Tiber based plywood and pre-laminated particle boards.

II. SCHEME DESIGN

A. PT Plan Documentation

PT provider is required to document a plan before the commencement of the scheme. The plan has to include the objectives, purpose and basic design of the PT scheme, including the following essential information:

❖ Selection of the measurand(s) or test parameters;
❖ A description of the range of values or characteristics;
❖ The potential major sources of errors;
❖ Requirements for the production, quality control, storage and distribution of proficiency test items/samples;
❖ Assurance of confidentiality;
❖ Reasonable precautions to prevent collusion between participants or falsification of results;
❖ Procedures for the test or measurement to be used for homogeneity and stability testing of proficiency test.

B. Statistical Design

Statistical design is required to be developed to meet the objectives of the scheme. PT Provider has to give careful consideration to the following items in designing statistical analysis:

❖ The accuracy (trueness and precision) as well as measurement uncertainty required or expected for each parameter;
❖ The relevance of significant figures to the reported result, including the number of decimal places;
❖ The number of proficiency test items to be tested or measured;
❖ Procedures used to establish the standard deviation for proficiency assessment (SDPA) or other evaluation criteria;
❖ Procedures to be used to identify or handle outliers, or both;
❖ Procedures for the evaluation of values excluded from statistical analysis.

C. Sample Preparation

The Proficiency Testing Programme has planned PT scheme on Ordinary Portland Cement-53 grade for Standard Consistency, 7-days Compressive Strength, and Soundness by Le-Chatelier tests as per IS 269-2015 with the relevant methods, that is, IS 4031(Part-4): 1988, IS 4031(Part-6): 1988 and IS 4031(Part-3): 1988 respectively. The PTP Division has secured OPC 53 grade in full bags in two numbers conforming to the requirements of IS 269-2015. The received bags are properly sealed and labeled with name, batch number, quantity, manufacturing date/month, and IS code. After verification they have been accepted for PT purposes. The PTP division hands over the OPC 53 grade bag to the Civil Engineering Division of NTH (WR) for making further it homogeneous by thoroughly mixing, coning and quartering on the line of IS 3535-1986 in dry condition and preparing the representative samples. PT item is forwarded to the 24 participant laboratories. After preparation of PT items, the Civil Engineering Division hands over the PT items to PTP Division where they are numbered, coded, and stored properly without any damage or deterioration. The randomly selected PT items are forwarded to Civil Engineering Division for conducting homogeneity and stability testing. Homogeneity and stability tests are conducted during the actual testing time .

III. HOMOGENEITY AND STABILITY TEST OF PT ITEM

As the homogeneity and stability of cement samples, which are used in the laboratory proficiency test, are the basis for performing the proficiency test plan, all cement samples must be completely well-proportioned, reliable & stable to ensure that any doubtful, unsatisfied results coming from the comparison result are not caused by the differences between the samples.

A. Homogeneity Test of PT Item: One of the most important prerequisite conditions for PT is to ensure the homogeneity of the PT items. Homogeneity of the samples is checked. The samples of 10 Nos. have been taken randomly for homogeneity tests. Duplicate test results are generated from each sample, thereby, 20 test results of each PT test parameter are generated. The between sample standard deviation of the test results is calculated and compared with the criteria set by the

TABLE I.
ASSESSMENT OF HOMOGENEITY

Parameters	Average of Homogeneity Test	Between Sample Standard Deviation (S_s)	Limiting Value ≤0.3*SDPA
Standard Consistency, %	29.80	0.118	0.3
Average Compressive Strength, N/mm^2	42.61	0.319	1.2
Soundness by Le- Chatelier method, mm	0.85	0.083	0.15

laboratory (≤0.3*SDPA). The result of the homogeneity checks clearly indicates that the samples are homogeneous. The details of homogeneity tests are shown in Table I.

B. Stability Test of PT Item : The stability of the samples is checked. The stability of the sample is verified in the regular interval of time during the testing period of the samples to ensure that the samples are stable throughout the testing time schedule of PT scheme. Total three samples have been taken randomly for stability tests. Each sample generated two results of each test parameter. Thereby, six test results are generated for each test parameter. The difference between the average values of stability test results and homogeneity test results are compared with the criteria ≤0.3*SDPA for all three test parameters. The result of the stability check clearly indicates that the samples are stable. The details of stability test are shown in Table II.

IV. REPORTING AND ANALYSIS OF DATA

The laboratories were advised to submit their measurement results on specially designed proforma given in the 'Technical Protocol'. They were also asked to submit copies of the calibration certificates for the instruments used in measurements for non NABL accredited laboratories. In this PT program all the participant laboratories have NABL accreditation. The values of measured parameters reported by participants are shown in Table III. Before compiling and comparing the results the following corrections are applied. The Assigned value and the uncertainty of the assigned value are evaluated by the Robust analysis Algorithm A, ISO 13528:2015. The participant's results are having metrological traceability and are in SI units. The performance of laboratories is evaluated by comparison with other participants. Initially the SDPA is chosen by perception. However, the acceptability of Homogeneity and Stability results are checked with SDPA from the analysis of Algorithm A of the participant's results. The trueness of the assigned value is verified as per Cl 7.8 of ISO 13528:2015. The results are found suitable and satisfactory.

V. STATISTICAL FINDINGS

24 laboratory results for Standard Consistency, Compressive Strength and 23 laboratory results for and Soundness by Le-Chatelier method have been statistically evaluated and Z scores are calculated. The Z score for the omitted test result (Soundness) of the participant laboratory is calculated separately. The

TABLE II.
ASSESSMENT OF STABILITY

Parameters	Average of stability Test	Average of homogeneity Test	Difference	Limiting Value ≤0.3*SDPA
Standard Consistency, %	29.75	29.80	0.050	0.3
	29.75	29.80	0.050	
	30.00	29.80	0.200	
Average Compressive Strength, N/mm^2	42.36	42.61	0.251	1.2
	43.01	42.61	0.399	
	42.18	42.61	0.436	
Soundness by Le- Chatelier method, mm	0.80	0.85	0.50	0.15
	0.80	0.85	0.50	
	0.88	0.85	0.025	

Table III.
RESULTS OF PROFICIENCY TESTING

S.No.	Participant Lab Code	Standard Consistency, %	Average Compressive Strength, N/mm^2	Soundness by Le-Chatelier method, mm
1	A	29.5	46.0	1.00
2	B	30.0	43.2	1.00
3	C	30.5	41.9	2.1$^\$$
4	D	30.5	45.5	0.50
5	E	30.0	45.6	0.40
6	F	28.5	51.2	0.70
7	G	30.7	46.8	0.50
8	H	31.0	38.4	-
9	I	30.0	51.5	0.50
10	J	30.5	44.5	1.00
11	K	31.0	40.1	1.00
12	L	30.0	40.5	1.00
13	M	31.0	40.1	1.00
14	N	31.0	40.3	1.00
15	O	29.0	46.1	0.50
16	P	29.0	46.5	0.50
17	Q	28.8	50.9	0.70
18	R	30.0	44.0	0.50
19	S	31.0	40.5	1.00
20	T	29.5	46.0	0.50
21	U	28.5	47.0	0.50
22	V	30.5	43.5	0.50
23	W	29.0	43.0	1.00
24	X	30.5	44.5	1.00

Table IV.
STATISTICAL FINDINGS

Parameter	Standard Consistency,%	Average Compressive Strength, N/mm2	Soundness by Le-Chatelier method, mm
No. of Labs. (N)	24	24	23
Minimum	28.50	38.4	0.4
Maximum	31.00	51.5	1.0
Average	30.00	44.48	0.74
Assigned Value	30.006	44.333	0.741
SDPA	0.938	3.687	0.284
Uncertainty of Assigned Value	0.239	0.941	0.074

details are given in table IV.

VI. Z-SCORE EVALUATION

The results are evaluated as per ISO 17043:2010 and Cl. 9.4 of ISO 13528:2015 by calculating the Robust Z Score of the test parameter of each participant laboratory. One participant test result for Soundness is omitted as it is outside the limits of the set criteria prior to the statistical analysis. However, Z score of the participant

lab result is calculated separately and reported. The procedure for Blunder removal NTH/WR/WP/QM-PTP/4.4/23 is followed for this purpose.

Performance evaluation for Test

Performance is evaluated by calculating Z score 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;
$$Z_i = (x_i - x_{pt})/\sigma_{pt}$$
where,
x_i is the test result from participant laboratory,
x_{pt} is the assigned value and
$_{pt}$ is the standard deviation for proficiency assessment (SDPA).
The assigned value $u(x_{pt})$ is calculated as follows;
$$u(x_{pt}) = 1.25s^*/\sqrt{p}$$
where,
s^* is robust standard deviation and
p is number of participants

As per ISO 13528:2015, performance of the laboratories with $|Z| \leq 2.0$ is considered satisfactory. The laboratories getting $|Z| \geq 3.0$ are considered outlier and those getting $2.0 < |Z| < 3.0$ score are considered as questionable performers.

VII. RESULT AND DISCUSSION

The test results which are received according to the time schedule and adhered to the instructions of testing are analyzed. The Participant Laboratories are uniquely coded by alphabets between 'A' to 'X'. The results are evaluated as per ISO 17043:2010 and ISO 13528:2015. After elimination of one extreme value in case of Soundness, the test results of 23 laboratories are statistically evaluated. The Z-Score for the omitted laboratory result is calculated separately. In case of Standard Consistency and Compressive Strength tests, the 24 participant results are analyzed as no participant test result is varying more than the limiting set criteria, that is, three times the calculated standard deviation +/- average value of participant results. 24 laboratory results

Table V.
Z-SCORE OF PARTICIPANT LABORATORY

S.No.	Participant Lab Code	Standard Consistency	Average Compressive Strength	Soundness by Le- Chatelier method
1	A	-0.54	0.45	0.91
2	B	-0.01	-0.31	0.91
3	C	0.53	-0.66	4.79[$]
4	D	0.53	0.32	-0.85
5	E	-0.01	0.34	-1.20
6	F	-1.61	1.86	-0.14
7	G	0.74	0.67	-0.85
8	H	1.06	-1.61	-
9	I	-0.01	1.94	-0.85
10	J	0.53	0.05	0.91
11	K	1.06	-1.15	0.91
12	L	-0.01	-1.04	0.91
13	M	1.06	-1.15	0.91
14	N	1.06	-1.09	0.91
15	O	-1.07	0.48	-0.85
16	P	-1.07	0.59	-0.85
17	Q	-1.34	1.78	-0.14
18	R	-0.01	-0.09	-0.85
19	S	1.06	-1.04	0.91
20	T	-0.54	0.45	-0.85
21	U	-1.61	0.72	-0.85
22	V	0.53	-0.23	-0.85
23	W	-1.07	-0.36	0.91
24	X	0.53	0.05	0.91

Table VI.
QUESTIONABLE PERFORMANCE

Parameter	No. of participant laboratory	No. of Questionable performance (2<|Z|<3)	No. of Outlying performance (|Z|≥3)
Standard Consistency,%	24	Nil	Nil
Average Compressive Strength, N/mm^2	24	Nil	Nil
Soundness by Le- Chatelier method, mm	23	Nil	1
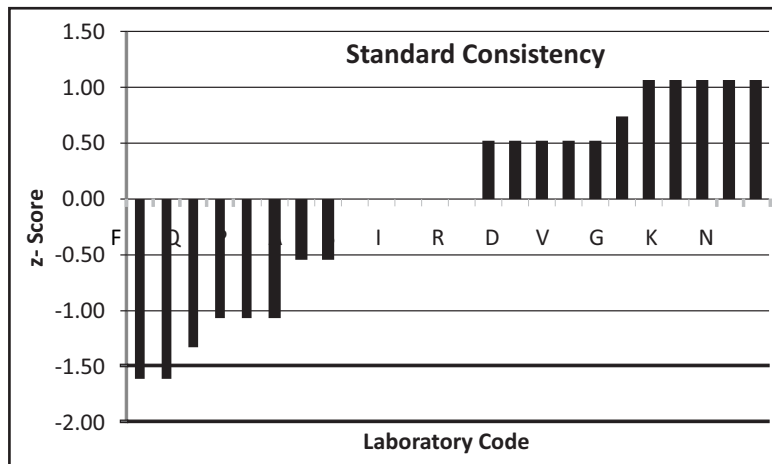
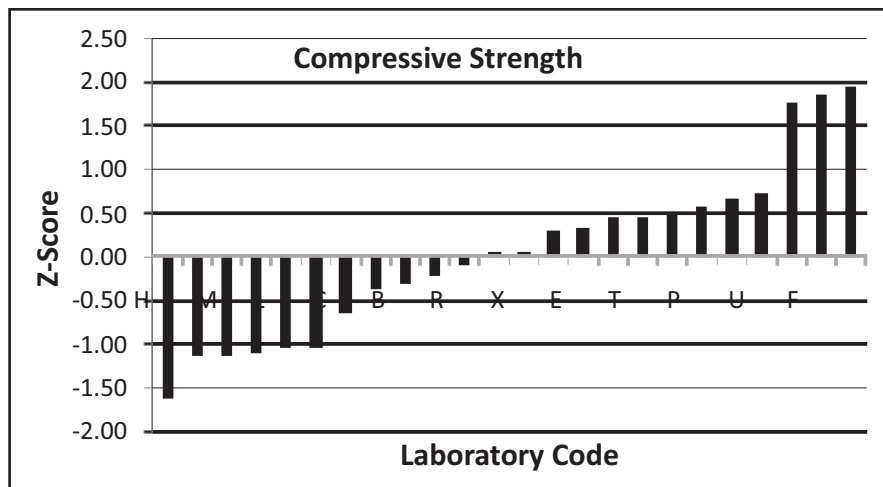**Fig. 1. Bar Chart showing the *z*-score for Standard Consistency Test**

Fig. 2. Bar Chart showing the *z*-score for Compressive Strength (7 days) Test
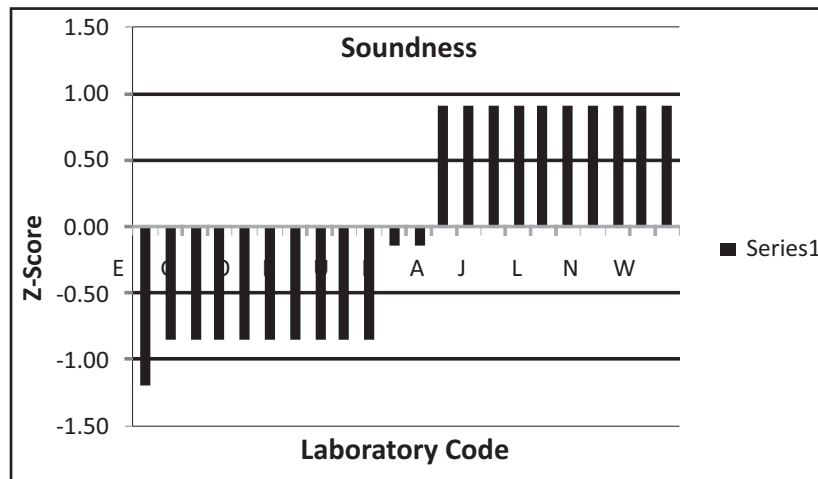
Fig. 3. Bar Chart showing the *z*-score for Soundness Test

for standard consistency, compressive strength, and 23 laboratory results for Soundness by Le-Chatelier method have been statistically evaluated and Z scores have been calculated. The Z score for the omitted test result (Soundness) of the participant laboratory is calculated separately. Participant laboratories Z Scores along with their results are given in Table V. The questionable performance of the laboratories is with (*) mark and the outliers are marked with (#). However, in the present scheme there are no questionable and outlier results. The data eliminated for the statistical analysis due to gross errors in the result is indicated by ($). Fig. 1 to 3 show the bar charts of Z scores.

VIII. CORRECTIVE ACTION AND SUGGESTION TO THE LABORATORY

As mentioned in section 6, the laboratories getting $|Z| \geq 3.0$ are considered outlier and those getting $2.0 < |Z| < 3.0$ score are considered as questionable performers requiring investigations and corrective action by the participating laboratory. The laboratory's management needs to ensure that the problem is rectified and procedures are put in place to prevent a recurrence. Laboratory with Code No. C was asked to review the result for soundness test and take appropriate corrective actions. This laboratory was requested to improve its testing facilities, to modify the measurement method, and to estimate the measurement uncertainties properly.

IX. CONCLUSION

The proficiency testing concludes that out of the total 72 measurement results reported here in this paper, 71 (98.6 %) are in agreement with the nodal laboratory. The z-score of only one laboratory (Lab Code-C) for soundness test are outside the acceptable limit. Overall, the results are considered to be excellent. The PT programme is organized completely in conformance with the international standard ISO/IEC 17043-2010 and ISO 13528-2015. Strict confidentiality is maintained in the PT scheme. The PT scheme is coordinated by the responsible group of Officers under the supervision of PTP Coordinator. The PT results and names of the participant labs of the scheme are kept under strict confidence and will not be disclosed to any person or to any organization, except to the Authoritative body (NABL) if asked for, without the prior approval of the concerned participant laboratory.

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