PEROFORMANCE REPORT

XXXIII ANALYTICAL QUALITY CONTROL PROGRAM

ON

TRACE METAL PARAMETERS

JULY 2021

LAB CODE	128

CENTRAL POLLUTION CONTROL BOARD

MINISTRY OF ENVIRONMENT, FOREST AND CLIMATE CHANGE

PARIVESH BHAWAN, EAST ARJUN NAGAR

DELHI – 110032

Background

The Instrumentation Laboratory of Central Pollution Control Board, Delhi has organized the **33rd Analytical Quality Control (AQC) exercise** during 2020-21 for the Environmental Laboratories of Central and State Pollution Control Boards, Pollution Control Committee's, Public Sector and EPA Recognized Private Laboratories for trace metal parameters. The purpose of the AQC program is to i) Continuously monitor the accuracy and reliability of the analytical data generated by the participating laboratories, ii) Help the laboratories to improve their infrastructure and iii) Enhancement in the quality of the laboratory generated analytical data.

Central Pollution Control Board (CPCB), Delhi has initiated this AQC exercise since 1991 with an aim to get reliable data from the laboratories, who have been involved in some important water quality monitoring programs/ projects under Global Environmental Monitoring System (GEMS), National Water Quality Monitoring Program (NWQMP), National Mission for Clean Ganga (NMCG) etc. in collaboration with State Pollution Control Board (SPCB)/ Pollution Control Committees (PCC). Initially, this program was started with participation of only 20 numbers of environmental laboratories. In past 30 years, CPCB had conducted 32 AQC exercises with near about 250 environmental laboratories from SPCBs and PCCs, other public sector and EPA recognised private laboratories. The last 32nd AQC exercise was conducted during 2017 for physico-chemical parameters.

The 33rd AQC exercise has been conducted amongst the 146 environmental laboratories across the country. CPCB laboratory has dispatched the samples to 146 participating laboratories through Courier service during 28th December 2020. However, 143 environmental laboratories have managed to submit their test results within the prescribed time line.

Out of 146 participating laboratories, 24 of them are located at Ganga front states (i.e. Uttarakhand, Uttar Pradesh, Bihar, Jharkhand and West Bengal). These laboratories are involved in continuous monitoring of water quality of river Ganga and its tributary rivers/ Drains. This exercise can improve the overall performance of the laboratory and ultimately help to meet the objective of "National Mission for Clean Ganga" (NMCG).

CPCB has evaluated the test results received from the participating laboratories. Accordingly, the performance report of each participant has been prepared on the basis of z- Score. The score will help the laboratory to understand the competency, in terms of analytical activities, the robustness of instruments used for sample analysis and the expertise of manpower engaged in the laboratory, and also it will help in overall improvement of the laboratory infrastructure to achieve world class quality data for competing at national and international platform.

Thank you for your active participation and support in the 33rd AQC program conducted such a period of Corona Virus diseases, COVID 19, pandemic. CPCB really appreciate the efforts of your laboratory to make the 33rd exercise a grant success.

1.0 Introduction

The Analytical Quality Control (AQC) exercise is designed to evaluate laboratory performance against other participating laboratories on the same set of environmental parameters. It is one of the major part of a quality assurance system (QA), wherein the quality of analytical data being generated in the laboratories is controlled through minimizing or controlling errors to achieve a target of accuracy. The correctness of the decision or action entirely depends upon the accuracy of the analytical results. If the errors of the analytical results were high, the man power, material and money spent on this study would be worthless and further lead to making wrong decision.

The implementation of standards set for discharge of effluent and emissions for industries and other sectors like Water Quality Monitoring (both Surface water and Groundwater), Ambient Air Quality etc. are significant for their compliance status. The monitored samples and their analytical reports are very much important where the authority could take the right decision in right time prior to any ecological loss. It is possible only when the laboratory provides accurate and reliable data. The quantitative analysis of samples through various environmental laboratories has played the key role. The laboratories have also achieved capacity to reach the desired depth of analysis as per environmental norms set for each type of industry for its discharge of effluent, emissions and sludge & other solid waste during the process. The performance of the laboratory could be evaluated through the exercises like Analytical Quality Control (AQC) program.

Laboratories can establish the competency of their testing services through routine participation in Analytical Quality Control (AQC) exercises based upon independent third party evaluation. This provides both the laboratory's customers and staff, the confidence in the analytical ability of the laboratory.

As per the "Guideline for Recognition of Environmental Laboratories under the Environment (Protection) Act, 1986, the Environmental Laboratories have to compulsorily participate in Analytical Quality Control exercise organised by the Central Government/ Central Pollution Control Board or an organisation designated to test the capabilities and integrity of the data generated by the recognised laboratories. The AQC performance of an individual laboratory is also taken care before granting consent for Recognition of Environmental Laboratories under the Environment (Protection) Act, 1986.

2.0 CPCB's 33rd AQC Exercise

Objectives of the 33rd AQC:

a. To evaluate the performance of the laboratory with respect to trace metal analysis.
b. To suggest, future course of action based on the AQC results.

   c.   Review for further renewal of EPA recognition for the laboratories aspiring for the same.

Programme Organization

The 33rd Analytical Quality Control (AQC) exercise has been organized for the environmental laboratories enlisted under E(P)A recognition and other interested laboratories for Trace Metal parameters during 2020-21. The contact details of the laboratories have been gathered and communicated them by sending letters/ emails to provide their consent for participation by paying the participation fee in CPCB's account with a time line. As per the consent received from the laboratories to participate in the 33rd AQC program, a list of participating laboratories has been finalized. Each of the participating laboratories was randomly allocated a unique code called laboratory code.

Preparation of Synthetic Samples for 33rd Analytical Quality Control exercise

The laboratory has prepared two AQC samples of different concentrations in bulk quantity for participating laboratories. These AQC samples were prepared with one lower concentration and another with higher concentration of the selected elements. The synthetic samples are prepared by using different concentration of single element Certified Reference Materials (CRMs) of each analyte. The AQC sample contains a mixture of selected heavy metals i.e. Total Arsenic, Cadmium, Total Chromium, Copper, Iron, Manganese, Nickel, Lead and Zinc at different concentrations. Suprapure Nitric acid is used as preservative while preparing the bulk samples to maintain pH at <2 and to minimize the cross contamination of the samples.

The AQC samples are then transferred into 250 ml volume, pre-labelled, high grade Polyethylene Plastic bottles for sending to the participating laboratories. The information provided on the labelled sticker includes the details of AQC Scheme, Round and Sample code etc. The sample code is also representing the laboratory code of respective participating laboratory.

Periodical Analysis of AQC Samples at Laboratory Site:

A part of the AQC sample was retained by the laboratory for their analysis at a regular time interval till the end of the time period assigned to participating laboratories. This analysis has helped to provide the parameter-wise information on the "Reference Value" for comparison and estimate the acceptable limits of the reported values.

Dispatch of AQC samples to the participating laboratories:

The sets of sealed AQC sample bottles (i.e. one having lower concentration and another is higher concentration) were packed in a paper box along with dry ice packets, sample instruction sheet and other packaging materials. These sample packets were dispatched to the participating laboratories through courier service with time line. Initially, the last date for submission of test result was February 05, 2021, which was extended after request received from some participating laboratories two more-week time period for submitting the 33rd AQC test

results due to COVID 19, pandemic. hence, the last date for submission of test result was extended up to 19th Feb, 2021.

Time Frame for Analysis of AQC Sample and Submission of Test Report:

A fixed time frame was given to the participating laboratories for the analysis of AQC samples i.e. January 11 to 22, 2021 to get an appropriate and justified result (the uniform loss of analytes if any, for each and every AQC sample in the due course of time). The participating laboratories have submitted their test reports as per the instruction enclosed with the AQC sample packet (i.e. Annexure – 1 & Annexure – 2). The test results were reported in mg/L and up to three digits after decimal. As instructed, the laboratories have sent their test report in a closed/ sealed envelope through Speed post/ Registered post/ Courier services.

Instructions to Participants:

Detailed instructions were given to participating laboratories in the form of "Instructions for Handling the Test Materials and Reporting the Results for the 33rd Analytical Quality Control (AQC) Exercise" and "Data sheet for reporting the analysis result of 33rd AQC samples". Participating laboratories should report a single result for each method they use to analyse the sample. It is essential that they must report their results as per the instructions specified in the result reporting format (i.e. Annexure – 2).

If the participant leaves a reported value blank, it is assumed that the laboratory did not test for this analyte and a "Not Reported" evaluation is given. The results reported with less than (<), Not Traceable (NT), Below Detection Limit (BDL) values etc. shall not be considered for evaluation and hence no z – score shall be given to them. These results are reported inappropriately and breachingthe result format. Thus, these parameters will be considered as not reported/ wrongly reported.

Compilation of the Test results:

The participating laboratories have submitted their test reports within the stipulated time period. The information received from the laboratories are compiled and the test results are inserted in the Excel format for their evaluation through Robust Statistics.

Evaluation Process of the Test results using Robust Statistics:

The evaluation procedures are based on Robust Statistics (as per calculation of z – score) to assess participant's performance. Robust statistics are statistics which are not highly influenced by the presence of extreme results. The "Classical" (and most commonly used) measure of the center a dataset, the mean (average), is not robust. A robust alternative to the mean is the median (the middle value).

The AQC exercise uses the consensus results provided by the participating laboratories in the round of testing to determine the acceptable range. Robust statistics use the median result rather than average. Use of the median minimizes any effects due to extreme (very high or very low) results and is observed to be a very fair way of assessing participant performance.

The laboratory-wise AQC results reported by the participating laboratories, were tabulated and summarized after the expiry of time limit for submission of results. The Median was calculated from the submitted AQC results and used as assigned value as per ISO 13528:2005. Variations within the laboratories and between the laboratories have been analyzed in terms of robust statistical method (Robust Z-score) for each laboratory and for each parameter to keep the results free from disturbances due to extreme value.

Robust statistical parameters used during the analysis of AQC Data are:

- Number of AQC results.
- Exclusion of invalid data
- Median.
- Inter Quartile Range (IQR).
- Normalized Inter Quartile Range (NIQR).
- Robust Z-score.

Number of AQC results

In 33rd AQC exercise, 143 numbers of participating laboratories have submitted their test reports. These reports are further considered for evaluation.

Exclusion of invalid data

Few participating laboratories have reported their AQC results with extreme value or rouge value, that too far from the consensus value (i.e. Median). These extreme results are removed from the list prior to the final evaluation of test results as per the international protocol. Due to calculation error, using wrong unit of measurement and typographical error etc. some laboratories have reported their results with extreme value. These results which are out of range of the Median ± 5 SD are considered as invalid data and removed from the list before their final evaluation.

Median

The median is the middle value of the group, i.e., half of the results are higher than it and half of the results are lower. If N (no of laboratories) is an odd number, then the median was determined as a single central value i.e. (N+1)/2. If it is an even number, then the median was calculated as the average of two central values [N/2+{(N/2) +1}]/2.

Inter Quartile Range (IQR)

Inter Quartile Range is the range between the third (Q3) and first (Q1) quartiles.

IQR=Q3 - Q1 i.e., (Third Quartile - First Quartile)

First Quartile $Q1 = \dfrac{(N+3)}{4}^{th}$ Value of sorted data.

Third Quartile $Q3 = \dfrac{(3N+1)}{4}^{th}$ Value of sorted data.

Normalized Inter Quartile Range (NIQR)

The normalized IQR is a measure of the variability of the results. It is equal to the Inter Quartile Range (IQR) multiplied by a factor of (0.7413), which makes it comparable to standard deviation.

1 Standard Deviation (NIQR) = IQR×0.7413

Robust Z-score

The Z-score is a measure of the deviation of the result from the assigned value and it has been calculated from the various statistical parameters detailed above.

Robust Z-Score = $\dfrac{\text{Reported Result – Median}}{\text{Standard Deviation (NIQR)}}$

Where

NIQR = IQR × 0.7413

IQR = Q3 -Q1 i.e., (Third Quartile - First Quartile)

3.0 Proficiency Testing / Analytical Quality Control Results Evaluation

It is typical in Analytical Quality Control Programs to highlight the results with an absolute Z-Score. The Z-Score of ± 2 is generally considered as Satisfactory, while the value is within ± 3 is known as Questionable and beyond ± 3 is considered as Unsatisfactory.

As such, the following absolute Z-score ranges have been used for assessment of performance of the laboratories.

Sl. No.	'z' Score	Performance Evaluation	Result	Remark Value
1.	± 2.0	Satisfactory	Pass	Y (Yes)
2.	± 3.0	Questionable	Fail	N (No)
3.	Beyond ± 3.0	Unsatisfactory	Fail	N (No)

For calculation of overall percentage performance of the laboratory, each satisfactory Z-score i.e. Y (Yes) value in the remark column was assigned the value score of 5.555% while each Unsatisfactory and Questionable Z-score i.e. N (No) value in remark column was assigned value Score zero (0). The total numbers of satisfactory Z-score or Yes values were multiplied with a score of 5.555 percent, to derive overall performance of participating laboratory, in percentage.

4.0 General suggestions to Enhance Laboratory Analytical Quality Performance

The following suggestions are provided to laboratories for improving the analytical capability and continuously achieving accuracy of analytical data:

- The laboratories, which could not be qualified for any of the parameters (Z score > 2.0), should give much attention for rectifying analytical problems associated with the concerned parameter, will have to undertake '**Root Cause Analysis and corrective action'**.

- Internal AQC system is to be strengthened in all the laboratories at a regular interval. Improvement in Internal AQC system is to be made with reference to selection of method, quality of chemicals and glassware, analytical balance, preparation of control charts etc.

- The laboratories should be equipped with latest models of instruments and equipped with advanced features.

- The laboratories should have Type -1, reagent water for trace metal analysis. Preferably, Certified Reference Material (CRM), high purity grade chemicals including suprapure/ ultrapure Nitric acid etc. have to be used for analytical activities.

- The laboratory analysts have to be periodically trained in methods of testing, handling and calibration of instruments etc. to improve the analytical performance.

- The laboratories have to ensure periodical calibration and standardization of instruments to generate good analytical results. The other quality control studies like Spike and Recovery method may be used to achieve good quality of results.

- The laboratory to have in-house / outside training on Analytical Quality Control for better accuracy of analysis. Laboratories should use known standards / certified reference materials for each parameter to standardize the methodology and check the results in order to confirm correctness of analysis.

- It is essential to find out the constraint faced by the laboratory analyst during the analysis individually and strategies are to be evolved for improvement.

- The laboratory should arrange training programs for their staff for upgrade their knowledge as well as advance techniques used for sample analysis.

Instructions for Handling the Test Materials and Reporting the Results for the 33rd Analytical Quality Control (AQC) Exercise

GENERAL INSTRUCTIONS

Please read the following instructions carefully before starting analysis of AQC samples.

Two numbers of synthetic water samples (A & B) of 250 ml. each labeled with laboratory code and sample code are provided for analysis of metals/ metalloid i.e. Total Arsenic (As), Cadmium (Cd), Total Chromium (Cr), Copper (Cu), Iron (Fe), Manganese (Mn), Nickel (Ni), Lead (Pb) and Zinc (Zn). Both A & B are to be analyzed separately for all 9 parameters.

A. Instructions for sample storage, processing, analysis and result reporting:

1. **Please inform us immediately in the following address, after the receipt of the Parcel of AQC samples at your laboratory.**
   bkj93.cpcb@nic.in, Mobile No. (WhatsApp No.) - 09717460998

2. All samples should be stored in a refrigerator at 2-8^{0}C from the time of arrival at your laboratory.

3. The AQC samples shall be analyzed directly, as received. **No digestion is required**.

4. Any dilutions made during sample analysis should be corrected before the submission of final results.

5. All samples should be equilibrated at room temperature 25 (± 2^{0}C) prior to any dilutions or analysis.

6. Samples should be analyzed by the normal methods used for those determinants by your laboratory. However, the method is to be mentioned in the result reporting format.

7. The unit of the reported results should be mg/L (Milligram per Litre). The acceptance of the reported results should be up to 3 digits after decimal (e.g. 1.123 mg/L).

8. Laboratory should report their analysis results as per the given format i.e. Annexure – 1. Kindly avoid the use of any separate result format.

9. Be sure that the laboratory code and sample code are properly mentioned in the result format while sending the report.

B. **Time period for sample analysis and reporting of results:**

1. The analysis of the AQC samples should be performed within the period from January 11 to 22, 2020 at your laboratory. Please inform us, if there is any deviation on the time period of sample analysis.

2. Laboratories should report the results in a closed and sealed envelope with captioned "**33RD AQC RESULTS**". The envelop should be sent through **Registered mail/ Courier service** in the following address:

   SHRI GURNAM SINGH
   ADDITIONAL DIRECTOR AND DIVISIONAL HEAD
   INSTRUMENTATION LABORATORY
   CENTRAL POLLUTION CONTROL BOARD
   PARIVESH BHAWAN, EAST ARJUN NAGAR
   SHAHDARA, DELHI – 110032.

   No other means of communication (i.e. email, WhatsApp etc.) regarding the submission of AQC results will be accepted.

3. **The last date for receipts of 33rd AQC results in CPCB, Delhi is February 05, 2021 at 5.30 PM.** No results will be entertained after this time period.

C. **Information regarding the concentration of analytes in the AQC samples:**

Sl. No.	Parameters	Maximum Concentration for Code A samples (mg/L)	Maximum Concentration for Code B samples (mg/L)
1.	Total Arsenic	0.040	0.500
2.	Cadmium	0.025	1.000
3.	Total Chromium	0.100	2.000
4.	Copper	0.150	1.500
5.	Iron	0.500	1.500
6.	Manganese	0.200	1.000
7.	Nickel	0.050	0.800
8.	Lead	0.030	0.200
9.	Zinc	1.000	2.000

Annexure – 2

Central Pollution Control Board
Instrumentation Laboratory
PariveshBhawan, East Arjun Nagar, Shahdara, Delhi – 110032

33RD AQC	2020 – 21

LAB CODE	

Data sheet for reporting the analysis result of 33rd AQC samples.

01.	Name of the Organization	
02.	Corresponding address of the laboratory with PIN Code	
03.	Phone/ Fax No.	
04.	Contact person of the laboratory with designation	
05.	Mobile No.	
06.	Sample analyzed by (Name & Designation):	
07.	Date of receipt of sample	

RESULTS

Sl. No.	Parameters	Sample Code& Results (mg/L)		Method Adopted	Instrument used	Calibration# graph attached (Y/N)	Date of analysis
		A	**B**				
1.	Total Arsenic						
2.	Cadmium						
3.	Total Chromium						
4.	Copper						
5.	Iron						
6.	Manganese						
7.	Nickel						
8.	Lead						
9.	Zinc						

#: A copy of the standard calibration graph of each analyte is to be attached with this result format.

Date: **Signature of the Laboratory Head**

	CENTRAL POLLUTION CONTROL BOARD
	CENTRAL POLLUTION CONTROL BOARD **Ministry of Environment, Forest & Climate Change** **Parivesh Bhawan, East Arjun Nagar** **Delhi - 110032**

33rd ANALYTICAL QUALITY CONTROL TESTING PROGRAM

Lab Code	128
Name	MANTEC CONSULTANTS PVT. LTD.
Address	D-36, Sector - 06, Noida-201 301 Mobile/ Phone No. : 0120 4215805, 8506993145
E mail id	manteclab@gmail.com

PROFICIENCY EVALUATION REPORT

Sl. No.	Test Parameter	Sample	Reference value (mg/L)	SD (NIQR Basis)	Lab's Reported Result (mg/L)	Lower Acceptable Limit (mg/L)	Higher Acceptable Limit (mg/L)	Robust Z - Score	Remark
1.	Total Arsenic (As)	A	0.020	0.004	0.019	0.013	0.027	-0.28	Y
		B	0.197	0.028	0.196	0.140	0.254	-0.03	Y
2.	Cadmium (Cd)	A	0.015	0.002	0.014	0.011	0.019	-0.45	Y
		B	0.500	0.037	0.533	0.426	0.573	0.91	Y
3.	Total Chromium (Cr)	A	0.059	0.008	0.056	0.043	0.075	-0.36	Y
		B	1.480	0.102	1.432	1.277	1.683	-0.47	Y
4.	Copper (Cu)	A	0.080	0.008	0.074	0.064	0.096	-0.73	Y
		B	0.799	0.041	0.769	0.717	0.881	-0.73	Y
5.	Iron (Fe)	A	0.399	0.036	0.396	0.327	0.471	-0.08	Y
		B	1.239	0.101	1.224	1.037	1.441	-0.14	Y
6.	Manganese (Mn)	A	0.102	0.010	0.093	0.082	0.122	-0.88	Y
		B	0.746	0.042	0.701	0.661	0.831	-1.06	Y
7.	Nickel (Ni)	A	0.039	0.004	0.041	0.030	0.048	0.45	Y
		B	0.403	0.023	0.394	0.357	0.449	-0.39	Y
8.	Lead (Pb)	A	0.020	0.003	0.020	0.014	0.026	0.00	Y
		B	0.104	0.014	0.099	0.077	0.131	-0.36	Y
9.	Zinc (Zn)	A	0.613	0.050	0.578	0.512	0.714	-0.69	Y
		B	1.020	0.060	1.022	0.900	1.140	0.03	Y
Overall Performance of Laboratory in %						**100.00**			

Note: Acceptable limit are calculated based on Reference value (Median) ± 2 Standard

Deviation (NIQR basis) i.e. within ± 2 Z Scores values for both sample A & B individually.

Abbreviations:

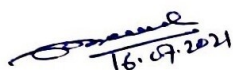NIQR = Normalized Interquartile Range

Z Score = (Lab's Reported Result – Reference value) / Standard Deviation (NIQR basis)

Y = Z Score within the acceptable limit

N = Z Score beyond the acceptable limit

NR = Data not Reported

WR= Wrongly Reported (Data not reported as per the prescribed format provided by CPCB, Delhi)

B. K. JENA
Scientist 'C'

NAMITA MISHRA
Scientist 'D'

GURNAM SINGH
Add. Director & Divisional Head,
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