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Technical Assistance
HYDROLOGY PROJECT

DHV CONSULTANTS & DELFT HYDRAULICS with HALCROW, TAHAL, CES, ORG & JPS

Findings of Third Inter-Laboratory AQC Exercise

Conducted by Central Ground Water Board – Bhopal

June 2002

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Foreword

Quality assurance and control is of prime concern in the generation of reliable water quality data. The Hydrology Project (HP) introduced during 1997-98, a two-tier system of analytical quality control (AQC) programme amongst the laboratories established/strengthened under the project. While the first one is for enhancing the precision in the data generation by a laboratory through 'within-laboratory' AQC exercise, the second is for minimizing the bias through 'Inter-laboratory' AQC exercise, so that the accuracy in data generation is maintained.

In the initial phase, the Central Pollution Control Board conducted two rounds of inter-laboratory AQC exercises amongst the HP-laboratories in December 1998 and April 2000. With a view to having continuity, the Ministry of Water Resources decided to build in-house capability for this purpose. Accordingly, the Central Water Commission's level II^+ laboratory at Hyderabad and the Central Ground Water Board's level II^+ laboratory at Bhopal have been selected to carry out the tasks in the surface and ground water domains respectively. The scientists/chemists of the selected laboratories have, therefore, been fully trained for this purpose. This will ensure sustainability, and gradual withdrawal of the external support. Accordingly, the two laboratories conducted the third round AQC programme during September-October 2001. The HP Consultants were also associated with the programme.

The present report is the outcome of the 3rd Inter-laboratory AQC exercises for the groundwater laboratories. Analysis of the AQC results was carried out using a user-friendly software developed under the project.

The Hydrology Project expresses thanks to the scientists/chemists of the Central Ground Water Board's laboratory at Bhopal for conducting the exercise and preparing the report in close co-operation with the HP-Consultants. The participating laboratories took keen interest in the programme.

We are confident that this annual activity will go a long way in generating reliable and useful data on a sustainable basis.

5 July 2002

J. G. Grijsen
Team Leader

1 Background

Analytical Quality Control **(AQC)** is one of the main components of a Quality Assurance system, wherein the quality of analytical data being generated in any laboratory is controlled through minimising or controlling errors to achieve a target accuracy. A particular water quality study or any organised water quality monitoring programme involves the collection, comparison and interpretation of analytical data, which leads to a decision for the management and use of the water resource. The correctness of decision or action depends largely upon the accuracy of the analytical results. If the errors in the analytical results are high, the manpower, material and money spent on any monitoring programme or study would be futile and further lead to wrong decision and improper action plans. Since the success and usefulness of an information system derived from analysis results depend on the quality of input data, it is essential to ensure that adequate comparability and accuracy of analytical results are achieved.

The need for analytical quality control (AQC) in HP laboratories was first discussed in the 'Water Quality Standardisation Workshop', December 9-10 1996, Hyderabad. It was recommended that AQC exercises should be organised for the HP laboratories to achieve a target accuracy, so that the results obtained from different laboratories are comparable. Consequently, the following actions were taken:

- In May 1997 and March 1998, many HP laboratories participated in 'within-laboratory AQC' exercises organised by HP consultants. The results of the first exercise were discussed in two technical meetings, held at Bangalore and Bhopal, and a report was brought out in February 1998. Results of the second exercise were reported in June 1999.
- Since the 'within-laboratory AQC' exercises evaluate only the precision of the laboratory, an 'inter-laboratory AQC' exercise was conducted in December 1998 by the Central Pollution Control Board (CPCB) to test the bias of the laboratories. Twenty-five (25) laboratories participated in this AQC-1 exercise.
- In continuation of the AQC-1 exercise, AQC-2 was conducted by CPCB in April 2000 for 42 laboratories.
- As a sequel to the AQC-1 and AQC-2 exercises, AQC-3 was conducted by the Regional Laboratory, Central Ground Water Board (CGWB), Bhopal, in September-October 2001 for 39 laboratories (mostly groundwater and combined surface and ground water laboratories). A comparable number of surface water laboratories also participated in AQC-3 was conducted by the Central Water Commission (CWC) Laboratory, Hyderabad. Both these laboratories, i.e. CGWB, Bhopal and CWC, Hyderabad, were trained at CPCB, Delhi for organising such AQC exercises under the guidance of the Hydrology Project (HP) Consultants.

For the 'inter-laboratory' AQC-3 exercise (groundwater), analysis of the data was performed by Dr. D. K. Goel, Scientist, CGWB, and the present report has been prepared by the HP-Consultants.

2 Objectives

The main objectives of an *inter-laboratory* AQC programme are:

- to assess the status of analytical facilities and capabilities of concerned laboratories;
- to identify the serious constraints (random & systematic) in the working environment of the laboratories;
- to provide necessary assistance to the concerned laboratories to overcome the short comings in the analytical capabilities;
- to promote the scientific/analytical competence of the concerned laboratories to the level of excellence for better output; and

to enhance the internal and external quality control of the concerned laboratories.

3 Methodology

3.1 Standard Samples

The list of parameters covered under the inter-laboratory AQC exercise is given below:

1. Conductivity (COND)
2. Chloride (Cl)
3. Total Hardness (TH)
4. Sodium (Na)
5. Fluoride (F)
6. Sulphate (SO_4)
7. Nitrate - N (NO_3 -N)
8. Phosphate-P (PO_4 -P)
9. Boron (B)

The above parameters were selected for the exercise because the procedures for their determination involve various analytical techniques viz. potentiometric (COND), titrimetric (TH and Cl), absorption-spectrometric (F, NO_3 -N, PO_4 -P and B), nephelometric (SO_4) and emission-spectrometric (Na) and common laboratory operations, such as weighing, heating, filtering etc. These tests are routinely carried out in the HP laboratories for characterisation of surface waters and groundwater. All HP laboratories are expected to be able to carry out these types of analyses. The capability of a laboratory to perform these tests satisfactorily would indicate its preparedness to carry out its assigned HIS functions.

Two synthetic samples, labelled as A & B, were prepared in the Regional Laboratory of the CGWB at Bhopal by mixing different volumes of 7 different solutions made from high quality chemicals and distilled water. The list of solutions, their strengths and the volumes used for each sample are given in Annexure I.

The samples were distributed to all the 38 participating laboratories through Courier service to avoid any delay in transportation of samples to laboratories. A copy of the communication indicating the instructions for carrying out analysis and the data reporting format are shown in Annexures II and IIa respectively. The original data received from the participating laboratories are presented in Annexure III.

3.2 Reference value

The parameter concentrations in each of the samples were determined in three different ways:

- from the stoichiometric considerations;
- from the chemical analysis in the CGWB-Bhopal laboratory; and
- from the combined results of analysis of the participating laboratories (reference mean).

The procedure for estimation of the reference value of a parameter from the combined analysis results of the participating laboratories is performed by the software developed by HP- Consultants for this purpose. Procedural steps for analysis of AQC data, as performed by the software, are outlined in Annexure IV.

Table 1 presented below compares the concentrations of the chosen parameters in the test samples, as estimated by the organising laboratory and referred to as the theoretical value. This value is the expert choice out of either the stoichiometric value or the analysis result with the values calculated from the combined results of analysis of the participating laboratories (reference mean). It can be seen that there is a close agreement among the values calculated by the two methods. Therefore, the reference mean is used for further data analysis.

S. No.	Parameter	Sample-A			Sample-B		
		Theoretical	Reference mean	Acceptable range	Theoretical	Reference mean	Acceptable range
1	Conductivity	310	317	307-328	380	382	365-400
2	Chloride	53	57	52-62	74	75	70-80
3	Total Hardness	70.0	68.2	65.4-71.1	90.0	91.3	86.1-96.4
4	Sodium	21.5	19.0	16-22	27.0	24.4	21.4-27.4
5	Fluoride	1.10	1.03	0.89-1.18	0.80	0.77	0.67-0.87
6	Sulphate	30.0	32.1	29.1-35.1	40.0	40.7	35.7-45.7
7	Nitrate-N	5.60	5.19	4.94-5.43	3.46	3.17	2.97-3.37
8	Phosphate-P	0.235	0.239	0.204-0.274	0.080	0.104	0.081-0.127
9	Boron	0.96	0.99	0.88-1.09	0.71	0.77	0.61-0.94

Table 1: Reference values and acceptable ranges for the AQC round ID: 3

3.3 Acceptable range for reported values

The acceptable limits for various parameters were arrived at adopting the method described in Annexure IV.

A laboratory was considered to qualify for the analysis of a parameter if both the reported analyses results for the two samples A and B lay within the acceptable ranges.

4 Findings and Discussions

4.1 Findings of AQC-Exercise

The reported analysis results for the two samples for each parameter were compared with the respective acceptable ranges shown in Table 1. The findings are summarised in Table 2, and are shown in the Youden 2-sample plots (Figures 1-9). The following observations can be made regarding the performance of the laboratories:

- Out of the 39 laboratories that participated in the AQC exercise, 38 laboratories have reported results (i.e. 1 laboratory did not send in results).
- Out of 38 laboratories responding, only 12 laboratories could provide results for all the 9 parameters.
- Out of 38 laboratories responding, the decreasing order of response for various parameters is as follows: Conductivity, Chloride and Total Hardness, 100%; Sodium, 97%; Fluoride and Nitrate 95%; Sulphate 89%; Phosphate-P, 68%; and Boron, 37%.
- Out of the 38 laboratories that reported, only 1 could analyse all the 9 parameters within acceptable limits. At the other extreme, 3 laboratories analysed only 1 parameter correctly.
- An overall view of the performance of laboratories for each parameter can be obtained from Youden 2-sample plots. Figures 1 to 9 give the plots for the 9 parameters covered under this exercise. For each parameter, the plot shows the value for Sample A against that for sample B reported by a laboratory. Thus, there is one data point for each laboratory. The acceptable limits for the two samples are also shown on the plot as two parallel horizontal lines for the sample values plotted on the Y-axis, and two parallel vertical lines for the sample values plotted on the X-axis. In case of plots for Sulphate, Nitrate-N and Phosphate, due to very high values reported by some of the laboratories narrowing down the acceptable range. This is causing overlapping of some of the points (please see Figures 6, 7 and 8). To get better resolution of the acceptable range and position of the laboratories, an additional plot is drawn for each of the above three parameters excluding the outliers, as shown in Figures 6a, 7a and 8a.

The centre of the rectangular block created by the two sets of parallel lines is the reference value for the parameter. Results close to this point are considered to represent a high degree of accuracy.

The figure can be divided in 4 quadrants by drawing a vertical and a horizontal line through the reference value. If only random error influences the determinations, the points would be randomly distributed in all the four quadrants. This is rarely seen. The points tend to concentrate in the first (++) or the third (--) quadrant, indicating that the laboratories tend to get either high values or low values, for both the samples. This points to the dominant role of systematic error. If a point lies on a line of unit slope passing through the reference value, then the determination has only systematic error.

Table 2: Performance of the laboratories in the AQC exercise round ID : 3

Lab ID	Laboratory Name	Cond	Cl	TH	Na	F	SO4	NO3-N	PO4-P	B	Total Reported	Within Limit	% success of reporting
101	CAP-HYD3	OK	OK	X	OK	X	OK	OK	X	-	8	5	63
102	SAP-HYD	OK	X	OK	OK	OK	X	X	OK	X	9	5	56
103	SAP-DOW1	OK	X	X	OK	X	X	X	X	X	9	2	22
104	SAP-CUD2	X	X	X	OK	X	-	X	-	-	6	1	17
105	SAP-KURN	X	OK	X	X	OK	-	X	-	-	6	2	33
106	CGU-AHM2	OK	X	X	OK	OK	OK	X	X	-	8	4	50
107	CGU-AHM1	X	OK	X	OK	X	X	OK	X	X	9	3	33
108	SGU-BHAV	X	X	OK	X	OK	OK	OK	OK	-	8	5	63
109	CKA-BAN2	OK	X	X	OK	X	OK	OK	X	X	9	4	44
110	SKA-BANG	OK	OK	X	OK	OK	OK	OK	-	-	7	6	86
111	SKA-BELG	OK	OK	OK	OK	X	OK	OK	-	-	7	6	86
112	SKA-BELL	OK	OK	OK	X	X	X	X	-	-	7	3	43
113	SKA-MYS2	OK	OK	OK	OK	X	OK	OK	-	-	7	6	86
114	CKE-THIR	OK	OK	OK	X	OK	OK	OK	X	X	9	6	67
115	SKE-THIR	X	X	OK	OK	X	X	X	OK	-	8	3	38
116	CMP-BHOP	OK	OK	OK	OK	OK	OK	X	X	OK	9	7	78
117	SMP-BHOP	X	X	X	X	OK	X	X	OK	X	9	2	22
118	SMP-SAGR	X	X	X	X	OK	X	X	X	-	8	1	13
120	SMP-SATN	OK	X	X	OK	X	X	X	X	-	8	2	25
121	SMP-BALG	X	OK	X	X	X	X	X	OK	-	8	2	25
122	SMP-GWAL	X	OK	X	X	X	X	X	X	OK	9	2	22
123	CMP-RAIP	X	OK	X	-	-	-	-	-	-	3	1	33
124	SMP-BILA	X	OK	X	OK	-	-	-	-	-	4	2	50
125	SMP-RAIP	X	OK	OK	X	X	OK	X	X	-	8	3	38

Lab ID	Laboratory Name	Cond	Cl	TH	Na	F	SO4	NO3-N	PO4-P	B	Total Reported	Within Limit	% success of reporting
126	CMH-NAG2	OK	OK	X	OK	OK	OK	X	X	-	8	5	63
127	SMH-KOKA	X	OK	X	OK	OK	X	X	-	-	7	3	43
128	SMH-AURA	OK	X	X	OK	X	X	X	-	-	7	2	29
129	SMH-NAS2	X	OK	X	X	X	OK	OK	-	-	7	3	43
130	SMH-PUN2	OK	X	X	OK	OK	X	OK	X	-	8	4	50
131	SMH-AMRA	X	OK	X	OK	X	OK	X	-	-	7	3	43
132	COR-BHU2	X	OK	OK	OK	X	OK	X	OK	OK	9	6	67
133	SOR-BHUB	OK	OK	X	X	X	X	X	X	X	9	2	22
138	STN-CHEN	OK	OK	OK	OK	OK	OK	OK	X	OK	9	8	89
139	STN-TRIC	OK	OK	OK	OK	OK	OK	OK	OK	OK	9	9	100
140	STN-MADU	OK	OK	X	OK	X	X	X	X	OK	9	4	44
141	STN-POLA	OK	X	X	OK	OK	OK	OK	OK	X	9	6	67
142	CTN-CHN2	OK	OK	X	OK	OK	OK	OK	OK	-	8	7	88
143	SGU-RAJ2	OK	X	OK	X	OK	OK	OK	OK	-	8	6	75
Labs reporting		38	38	38	37	36	34	36	26	14	297	151	51
No. Acceptable		22	24	13	25	17	19	15	10	6			
% success of reporting		58	63	34	68	47	56	42	38	43			
Ranking(of reporting labs)		3	2	9	1	5	4	7	8	6			

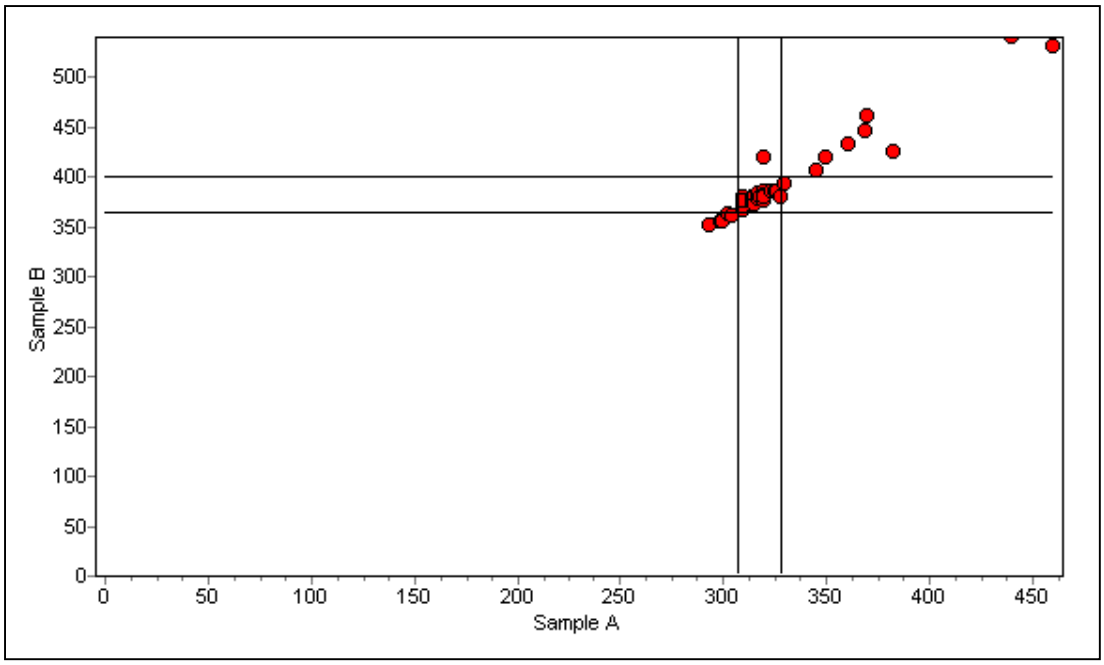

Figure 1: Performance of laboratories for conductivity-Youden 2 sample plot AQC Round ID: 3

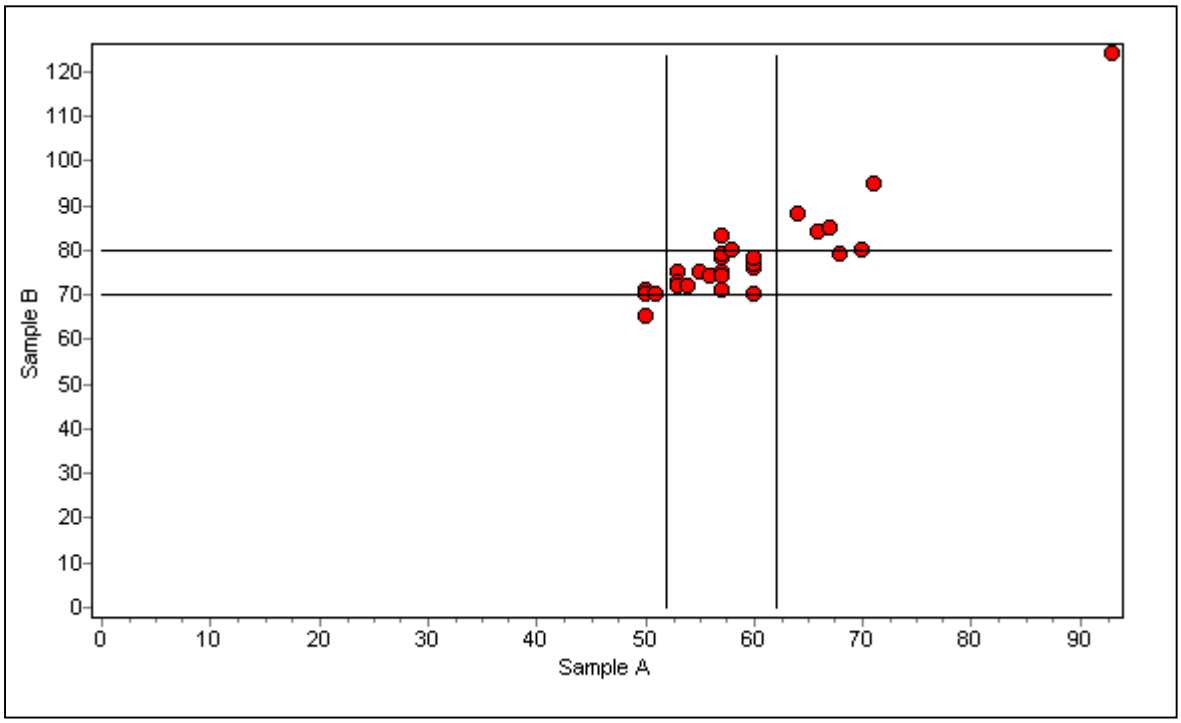

Figure 2: Performance of laboratories for Chloride- Youden 2-sample plot AQC Round ID: 3

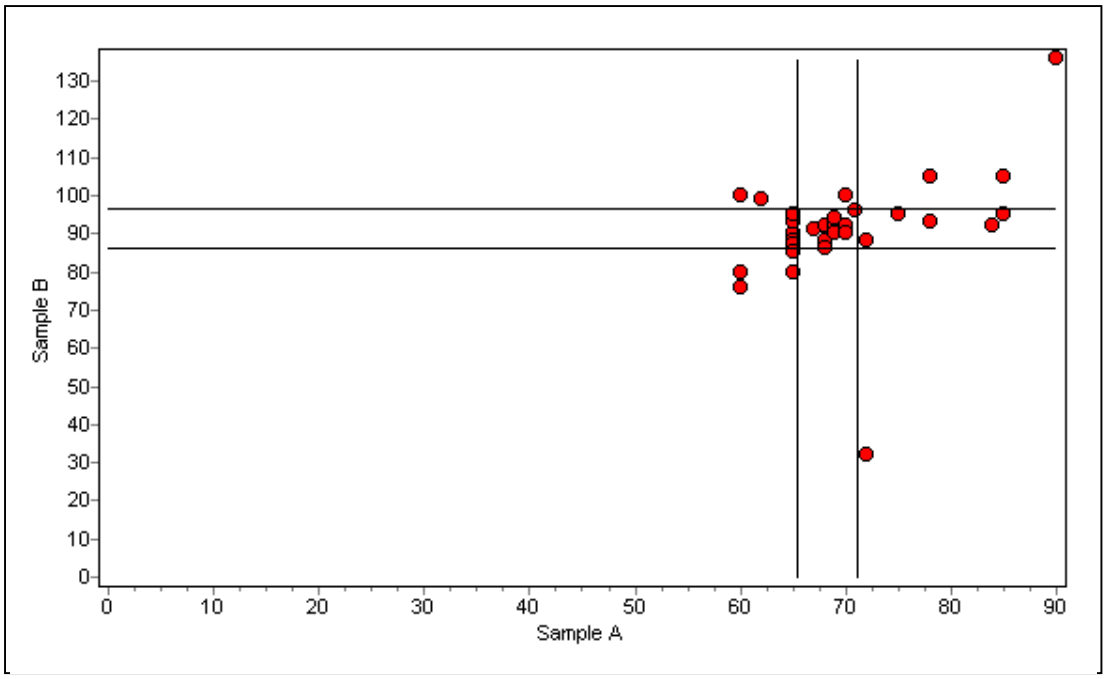

Figure 3: Performance of laboratories for Total Hardness-Youden 2-sample plot AQC Round ID: 3

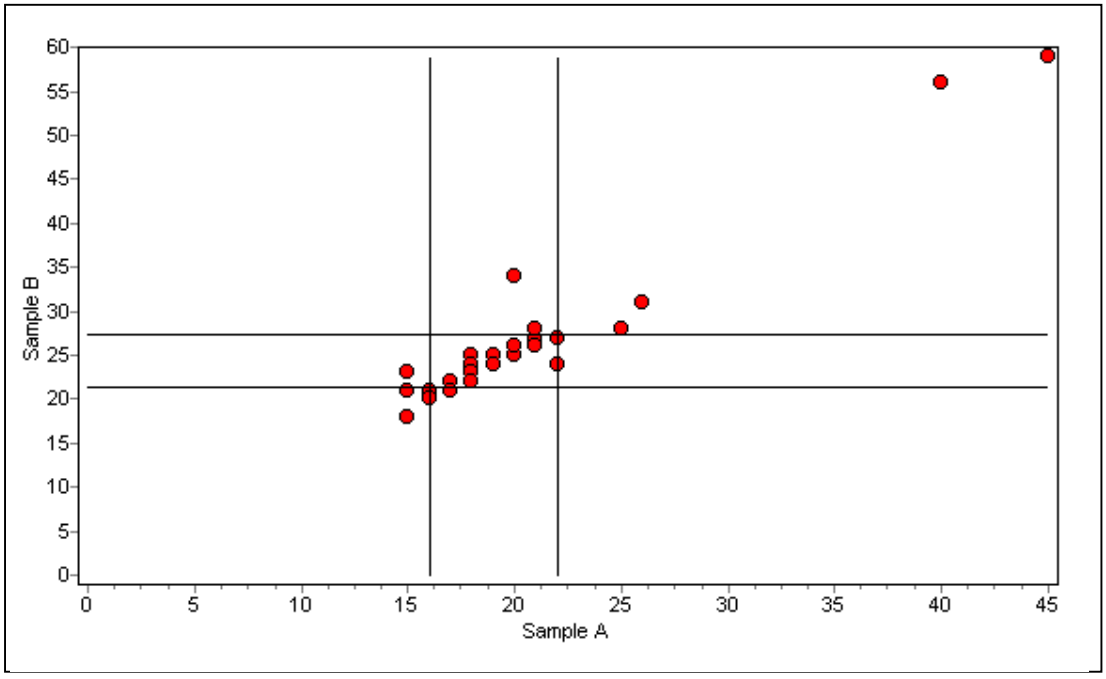

Figure 4: Performance of laboratories for Sodium-Youden 2-sample plot AQC Round ID: 3

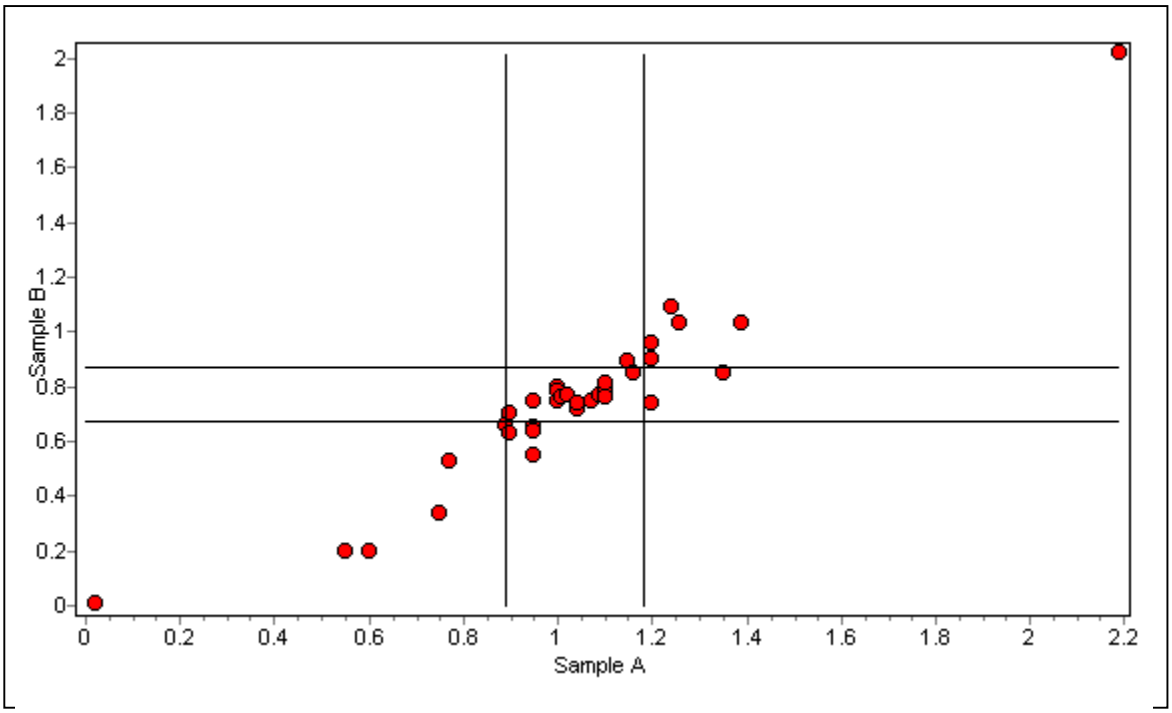

Figure 5: ***Performance of laboratories for Fluoride-Youden 2-sample plot AQC Round IS: 3***

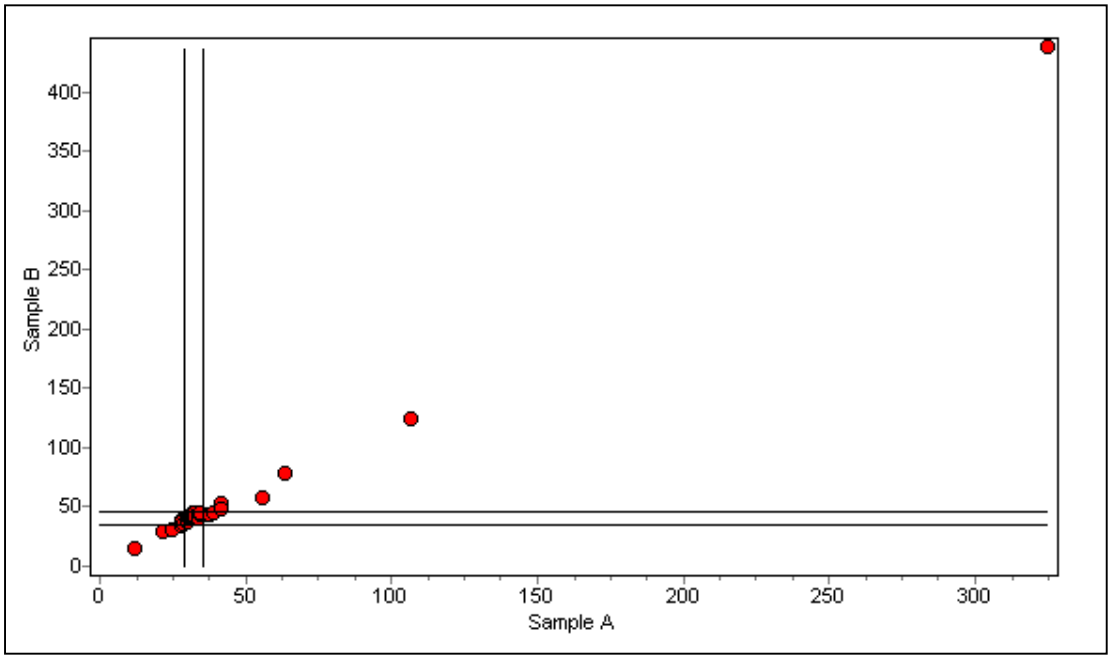

Figure 6: ***Performance of laboratories for Sulphate-Youden 2-sample plot AQC Round ID: 3***

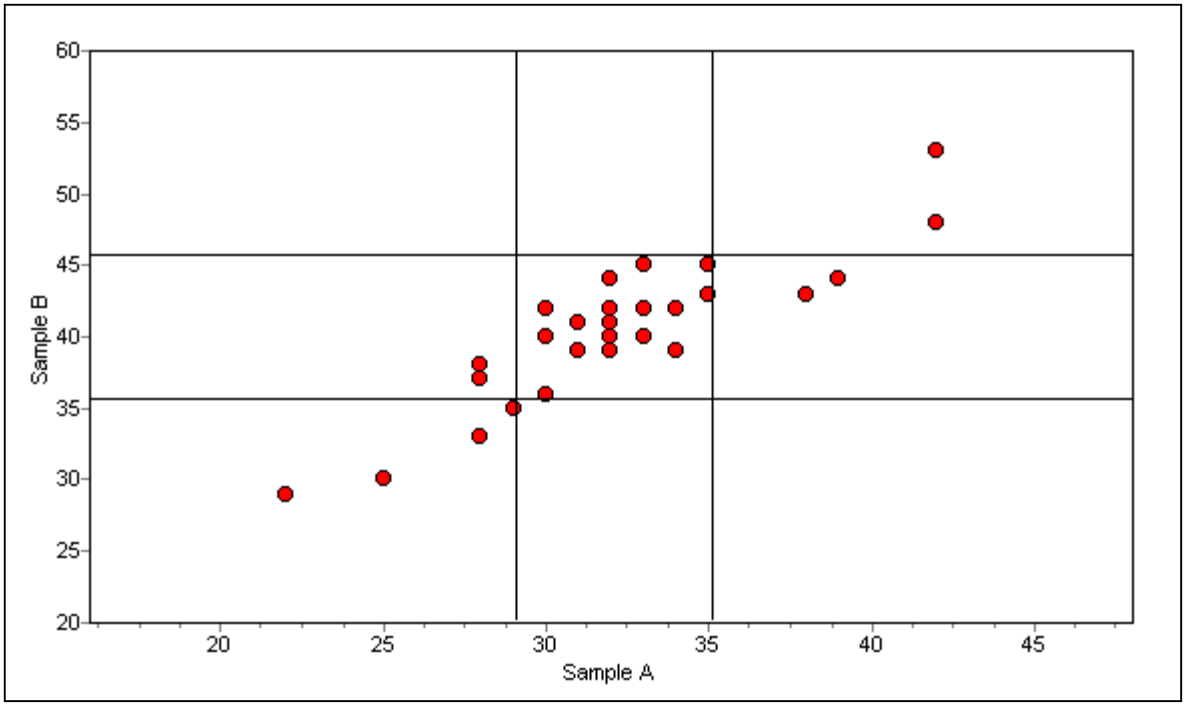

Figure 6a: ***Performance of laboratories for Sulphate-Youden 2-sample plot AQC Round ID: 3 (excluding outliers)***

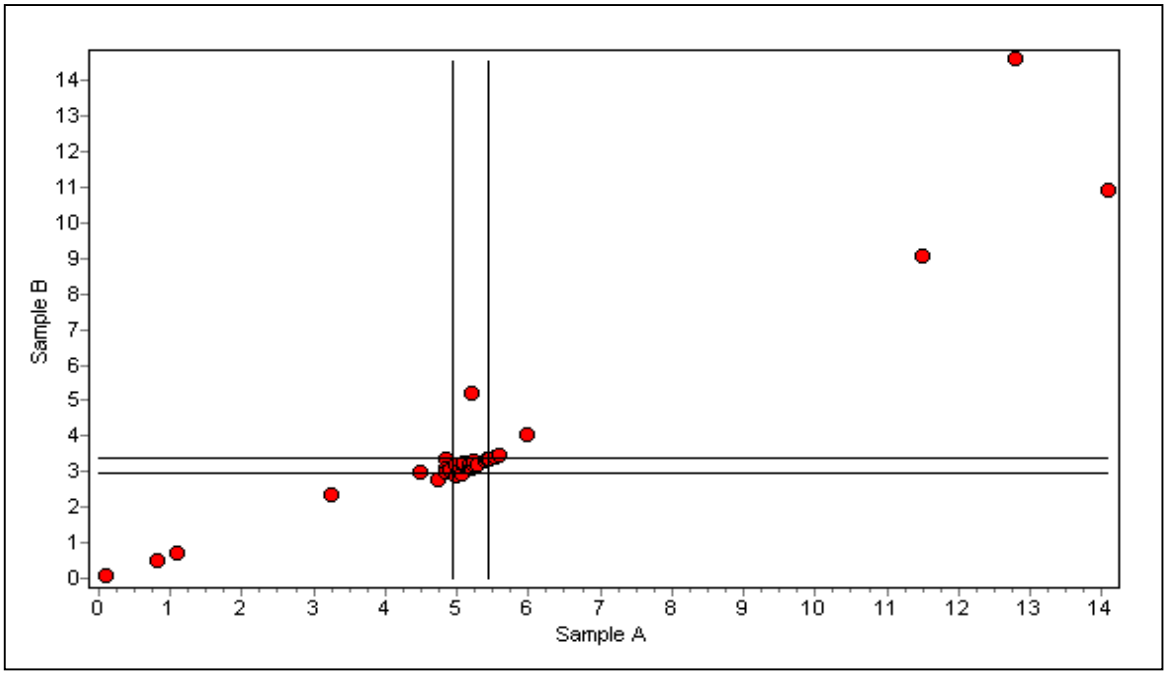

Figure 7: ***Performance of laboratories for Nitrate-N-Youden 2-sample plot AQC Round ID: 3 (all laboratories)***

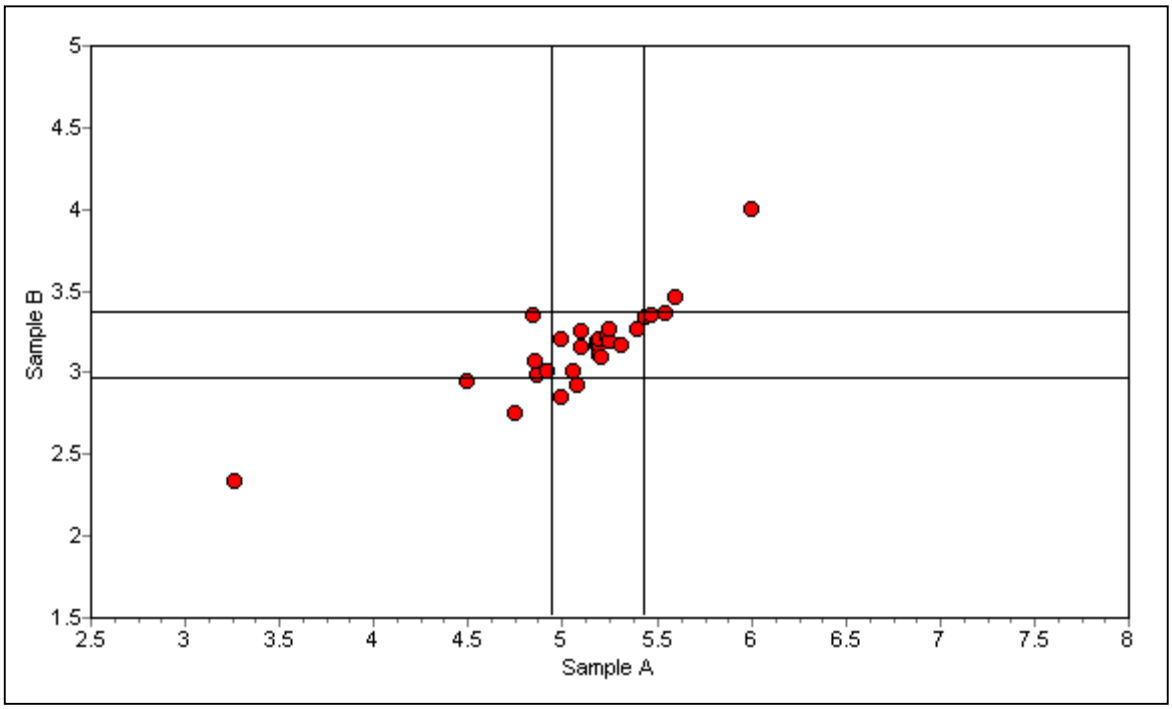

Figure 7a: Performance of laboratories for Nitrate-N-Youden 2-sample plot AQC Round ID: 3 (excluding outliers)

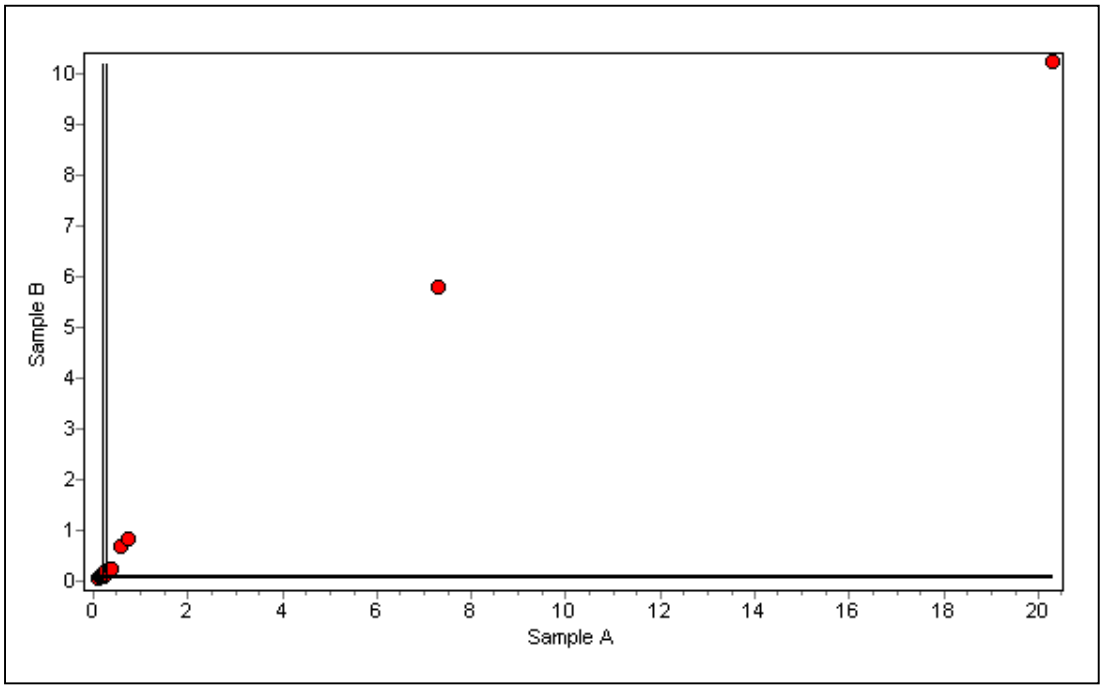

Figure 8: Performance of laboratories for Phosphate-P-Youden 2-sample plot AQC Round ID: 3 (all laboratories)

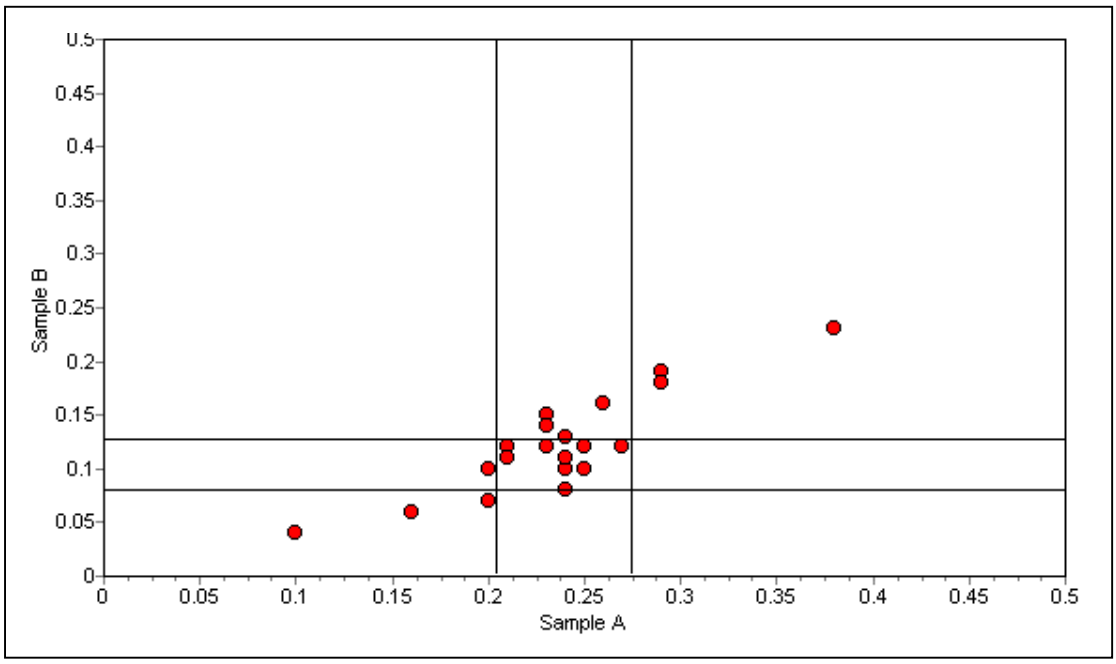

Figure 8a: Performance of laboratories for Phosphate-P-Youden 2-sample plot AQC Round ID: 3 (excluding outliers)

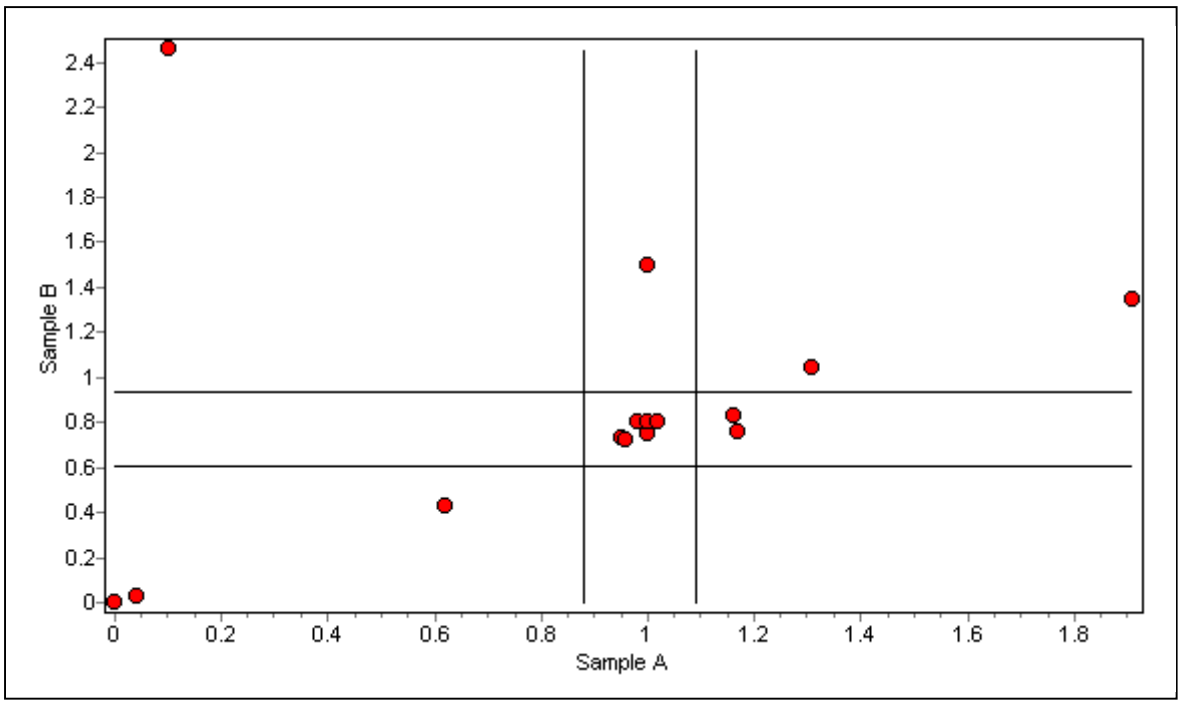

Figure 9: Performance of laboratories for Boron-Youden 2-sample plot AQC Round ID: 3

5 Conclusions and Recommendations

5.1 Conclusions

- The overall performance of the 38 laboratories participating in the AQC-3 exercise was not satisfactory. Only one laboratory could analyse all 9 parameters within acceptable limits. At the other extreme, 3 laboratories identified only 1 parameter correctly. However performance of CGWB lab and some state laboratories in MP has improved.
- The overall performance of the AQC-3 exercise reveals that many laboratories could not report correct results for many commonly measured water quality parameters. Specifically, many laboratories had difficulty in correctly analysing Phosphate and Boron.
- There was low response in the analysis of some parameters, especially boron. The reasons for this are not clear, since all laboratories should have the necessary analytical equipment and chemicals, and have received the 'Guidelines on Standard Analytical Procedures' (HP, 1999).
- There is still a need for laboratories to improve the quality of their analytical procedures so that meaningful data is being produced in laboratories.
- In the results the systematic errors prevail over the random error.

5.2 Recommendations

- Since overall performance in AQC-3 has marginally improved, it is essential to identify the problems affecting each laboratory individually and strategies are to be evolved for improvement. Laboratories which have not qualified for a parameter should give much attention for rectifying problems associated with the concerned parameter and should discuss issues with the HP consultants where there are uncertainties.
- In many cases, improvement in performance of laboratories can be brought about by selection of better grade chemicals, glassware, and distilled water. Analytical grade reagents (e.g. AR or GR) should be used for preparing all primary standards. All the laboratories should have good distilled water generation facility to have better quality and sufficient quantity of distilled water supply. This can improve the systematic error significantly.
- All laboratories should follow standard uniform analytical methods which are described in 'Guidelines for Standard Analytical Procedures' (HP, 1999) which is available at all laboratories. This will prevent unnecessary errors which may be caused in the calculation or reporting of results, as is suspected for some of the reported errors of nitrate.
- Internal AQC system with Shewart charts is to be introduced in all the laboratories on a regular basis to control random error, if not already introduced. Procedures for Quality Assurance and Within laboratory AQC are given in the Water Quality Training Module number 49. The use of blind samples may be considered in this context.
- It is utmost necessary to calibrate and standardise the instruments periodically to generate good analytical results. Different instruments have different requirements.
- It is suggested to have continuous and regular participation of AQC exercise for the participating laboratories in order to improve the analytical ability.

Annexure I
Composition of standard samples

CENTRAL GROUND WATER BOARD
Chemical Laboratory, North Central Region, Bhopal

INTER-LABORATORY ANALYTICAL QUALITY CONTOL EXERCISE
FOR THE
LABORATORIES OF GROUNDWATER DEPARTMENTS
OF
CENTRAL AND STATE AGENCIES UNDER

HYDROLOGY PROJECT

SEPTEMBER-OCTOBER, 2001

I. Preparation of stock solutions: 9-10 September 2001

S. No.	Name of chemical	Weight in g.	Final volume	Concentration
1	Magnesium Sulphate ($Mg\ SO_4.7H_2O$)	10.141	1 litre	1000 mg/L Mg 3952 mg/L SO_4
2	Calcium Chloride ($Ca\ Cl_2.\ H_2O$)	7.336	1 litre	2000 mg/L Ca 3538 mg/L Cl
3	Sodium Flouride (NaF)	1.105	1 litre	500 mg/L F
4	Potassium Nitrate (KNO_3)	7.218	1 litre	605 mg/L K 1000 mg/L NO_3-N
5	Boric Acid (H_3BO_3)	5.720	1 litre	1000 mg/L B
6	Potassium Dihydrogen Phosphate (KH_2P0_4)	0.439	1 litre	100 mg/L PO_4-P
7	Sodium Chloride (NaCl)	8.242	1 litre	3244 mg/L Na 5000 mg/L Cl

II. Preparation of liquid sample for analysis:

SAMPLE-A

400 mL $Mg\ SO_4.7H_2O$ + 350 mL $CaCl_2.2H_2O$ + 100 mL Na F + *300* mL KNO_3 + 50 mL H_3BO_3 + 150 mL KH_2P0_4 + 300 mL NaCl → Final volume 50 litres.

SAMPLE -B

500 mL $Mg\ SO_4.7H_2O$ + 500 mL $CaCl_2.2H_2O$ + 70 mL Na F + 200 mL KNO_3 + 40 mL H_3BO_3 + 100 mL KH_2PO_4 + 400 mL NaCl → Final volume 50 litres.

III. Constituents of liquid samples

Parameter	Sample-A	Sample-B
Sodium (Na), mg/L	310	380
Sulphate (SO_4) , mg/L	30	40
Fluoride (F) , mg/L	1.10	0.80
Nitrate-Nitrogen (NO_3-N) , mg/L	5.60	3.46
Boron (B) , mg/L	0.96	0.71
Phosphate-P (PO_4) , mg/L	0.235	0.080
Total Hardness (TH) , mg/L	70	90

Annexure II
Communication with the despatch of samples

CENTRAL GROUND WATER BOARD
Chemical Laboratory, North Central Region, Bhopal

INTER-LABORATORY ANALYTICAL QUALITY CONTOL EXERCISE
FOR THE
LABORATORIES OF GROUNDWATER DEPARTMENTS
OF
CENTRAL AND STATE AGENCIES UNDER

HYDROLOGY PROJECT

SEPTEMBER-OCTOBER, 2001

Please read the following instructions carefully before starting analysis of samples

Two nos. of synthetic water samples (A & B) of one lit. each labelled with lab code are provided for analysing Conductivity, Chloride, Total Hardness, Sodium, Fluoride, Sulphate, Nitrate-N, Phosphate-P and Boron.

Both labelled liquid samples (A & B) are to be analysed separately for all 9 parameters as shown below.

Important: Please report the results using the correct number of decimals, as specified in the table below.

- Conductivity should be reported in whole numbers (0 decimals) e.g. 256 μmhos/cm.
- Nitrate should be reported with 2 decimals, e.g. 5.17 mg N/L

S. No.	Parameter	Unit	No. of decimals to report
01	Conductivity at 25°C	μ mhos/cm	0
02	Total Dissolved Solids	mg/L	0
03	Total hardness as $CaCO_3$	mg$CaCO_3$/L	0
04	Sodium	mg/L	0
05	Fluoride as F	mg/L	2
06	Sulphate as SO_4	mg/L	0
07	Nitrate – N	mg N/L	2
08	Phosphate – P	mg P/L	3
09	Boron	mg/L	2

Note: Choose appropriate sample volume for each parameter for single run, so that analysis can be done within the provided sample volume.

ANALYTICAL METHODS:

1. You may choose any relevant method being followed in your laboratory for various parameters. However, the method is to be mentioned into the data format.
2. In case of colourimetric method, the standard graph and the factor used for calculation for sample analysis is to be submitted along with the data sheet.
3. Brief outline of the procedures for each analytical parameter is to be provided as Annexure along with the data sheets.

Please note the following points:

- All the samples are to be analysed most preferably during 18th September 2001 to 28th September 2001 for better comparison purpose of data obtained from various laboratories.
- Report the analysis result in the enclosed Data format sheet only. Kindly avoid using separate typed data sheet.
- Be sure that Lab code & sample code numbers are mentioned in the Data format sheet while sending the report.
- Be sure that all the units of various parameters are properly taken care of while reporting data. Especially important: Please be sure to report Nitrate in mg N/L and Phosphate in mg P/L.
- Analysis report should be sent directly to the following address positively latest by 15th October 2001 positively.

Dr. D. K. Goel,
Scientist C,
Central Ground Water Board,
North Central Region,
Block I, 4th Floor,
Paryawas Bhawan,
38 Arera Hills,
Bhopal-462 011

Annexure IIa

CENTRAL GROUND WATER BOARD

CHEMICAL LABORATORY, NORTH CENTRAL REGION, BHOPAL

III AQC / WATER	SEP 2001

LAB CODE	

III ANALYTICAL QUALITY CONTROL (AQC/WATER) EXERCISE - SEPTEMBER 2001 FOR THE LABORATORIES OF SURFACE AND GROUND WATER DEPARTMENTS OF CENTRAL AND STATE AGENCIES UNDER "HYDROLOGY PROJECT"

01	Name of the Organisation	
02	Address of the laboratory with PIN code, phone and fax	PIN …… Phone …….. Fax ……..
03	Sample analysed by: (Name & Designation)	1 …….. 2 …… 3 …….. 4 ……
04	Date of receipt of sample	

RESULTS

S. No.	**Parameter**	**Sample Code**		**Method adopted**	**Instrument used**	**Calibration Graph attached (yes/no)@**	**Date of analysis**
		A	**B**				
01	Conductivity at 25°C (µmho/cm)						
02	Chloride (mg/L)						
03	Total Hardness (mg/L)						
04	Sodium (mg/L)						
05	Fluoride (mg/L)						
06	Sulphate (mg/L)						
07	Nitrate-N (mg/L)						
08	Phosphate-P (mg/L)						
09	Boron (mg/L)						

@ A copy of the standard calibration graph, wherever applicable, is to be attached in Annexure.

Annex: III

Table 1: Original data for AQC Round ID: 3

Lab ID	EC_A	EC_B	CHLORIDE _A	CHLORIDE _B	Total HAR_A	Total HAR_B	Na_A	Na_B	F_A	F_B	SO4_A	SO4_B	NO3-N_A	NO3-N_B	o-PO4_A	o-PO4_B	B_A	B_B
101	323	385	55	75	65.0	90.0	19.0	25.0	0.90	0.63	33.0	40.0	5.20	3.10	0.160	0.060		
102	320	381	51	70	68.0	88.0	19.0	25.0	1.10	0.79	28.0	38.0	5.44	3.34	0.250	0.120	1.16	0.83
103	310	370	66	84	78.0	93.0	21.0	27.0	1.39	1.03	29.0	35.0	5.55	3.36	0.290	0.190	1.31	1.04
104	383	425	70	80	60.0	100.0	18.0	24.0	1.15	0.89			0.84	0.46				
105	370	460	60	70	60.0	100.0	25.0	28.0	1.04	0.74			1.10	0.67				
106	320	385	50	71	70.0	100.0	17.0	22.0	1.00	0.80	30.0	36.0	4.50	2.94	0.260	0.160		
107	300	355	60	76	62.0	99.0	19.0	25.0	2.19	2.02	22.0	29.0	5.24	3.21	20.320	10.230	0.10	2.46
108	299	355	50	70	69.0	92.0	16.0	21.0	1.07	0.75	32.0	42.0	5.19	3.19	0.240	0.100		
109	310	367	67	85	85.0	105.0	19.0	24.0	1.35	0.85	32.0	40.0	5.19	3.16	0.240	0.130	1.17	0.76
110	315	380	53	73	65.0	88.0	19.0	24.0	0.95	0.75	33.0	42.0	5.20	3.16				
111	314	374	53	73	68.0	88.0	19.0	24.0	0.95	0.55	35.0	43.0	5.06	3.00				
112	328	380	60	77	69.0	94.0	16.0	20.0	0.89	0.66	64.0	78.0	11.51	9.03				
113	310	370	53	73	68.0	88.0	19.0	25.0	0.95	0.65	31.0	41.0	5.10	3.15				
114	314	380	54	72	70.0	90.0	15.0	18.0	1.04	0.72	33.0	45.0	5.00	3.20	0.100	0.040	1.00	1.50
115	320	420	93	124	71.0	96.0	19.0	25.0	0.75	0.34	25.0	30.0	5.00	2.85	0.270	0.120		
116	310	380	53	75	70.0	90.0	22.0	27.0	1.10	0.80	30.0	40.0	5.60	3.46	0.240	0.080	0.96	0.72
117	330	393	50	70	65.0	80.0	20.0	34.0	1.10	0.81	28.0	37.0	4.92	3.00	0.210	0.110	1.91	1.35
118	460	530	71	95	90.0	136.0	45.0	59.0	0.90	0.70	107.0	123.0	12.80	14.60	0.750	0.820		0.00
120	326	386	68	79	68.0	86.0	19.0	24.0	0.02	0.01	325.0	438.0	0.11	0.07	0.580	0.680		
121	369	445	57	79	72.0	32.0	21.0	28.0	0.55	0.20	56.0	57.0	4.87	2.98	0.210	0.120		
122	440	540	58	80	84.0	92.0	40.0	56.0	1.24	1.09	12.0	15.0	4.75	2.75	7.300	5.800	1.00	0.75
123	350	420	57	78	75.0	95.0											0.00	
124	345	406	56	74	85.0	95.0	18.0	22.0										
125	361	433	57	74	68.0	92.0	15.0	23.0	0.60	0.20	35.0	45.0	4.86	3.07	0.200	0.070		
126	310	370	53	75	70.0	100.0	18.0	25.0	1.01	0.76	32.0	39.0	5.08	2.92	0.230	0.150		
127	304	360	60	78	65.0	88.0	18.0	22.0	1.00	0.78	39.0	44.0	6.00	4.00				
128	317	384	64	88	72.0	88.0	19.0	24.0	0.77	0.53	42.0	53.0	14.10	10.90				
129	302	362	57	71	60.0	76.0	17.0	21.0	1.20	0.74	34.0	42.0	5.20	3.20				
130	310	376	51	70	65.0	87.0	18.0	23.0	1.10	0.76	38.0	43.0	5.25	3.19	0.230	0.140		
131	293	351	60	78	60.0	80.0	22.0	24.0	1.20	0.96	32.0	44.0	4.85	3.35				
132	302	363	54	72	67.0	91.0	18.0	23.0	1.20	0.90	31.0	39.0	5.47	3.35	0.250	0.120	0.98	0.80
133	325	385	57	75	65.0	85.0	26.0	31.0	0.95	0.64	28.0	33.0	3.26	2.33	0.380	0.230	0.04	0.03

Lab ID	EC_A	EC_B	CHLORIDE _A	CHLORIDE _B	Total HAR_A	Total HAR_B	Na_A	Na_B	F_A	F_B	SO4_A	SO4_B	NO3-N_A	NO3-N_B	o-PO4_A	o-PO4_B	B_A	B_B
138	317	377	57	74	70.0	92.0	19.0	24.0	1.00	0.75	30.0	36.0	5.25	3.26	0.200	0.100	1.00	0.80
139	318	380	53	72	70.0	90.0	21.0	26.0	1.09	0.77	34.0	39.0	5.40	3.26	0.230	0.120	0.95	0.73
140	315	372	57	74	78.0	105.0	20.0	25.0	1.26	1.03	42.0	48.0	5.21	5.21	0.290	0.180	1.02	0.80
141	317	377	57	83	65.0	95.0	20.0	26.0	1.02	0.77	32.0	42.0	5.10	3.25	0.240	0.110	0.62	0.43
142	320	375	57	74	65.0	93.0	18.0	24.0	1.16	0.85	30.0	42.0	5.31	3.17	0.230	0.120		
143	320	380	50	65	69.0	90.0	15.0	21.0	1.07	0.75	32.0	41.0	5.21	3.09	0.250	0.100		

ANNEX IV
Procedural steps for Analysis of AQC data as performed by the software

Step	Procedure	Comments
1	Tabulate raw data for the laboratories (1st column Table 1 "all data")	Sorted by laboratory ID for easy reference. Displays: count, min, max, mean, stdev (for n-1 d.o.f), c.v.
For all samples (A and B) for each parameter (at present 9 numbers), the following steps are performed(*)		
2a	First Screening: remove data too far from theoretical value Tabulate remaining data (2nd column Table 1 "Xtheo")	Accept only data within a fixed predefined margin based upon the theoretical concentration value of the sample. Acceptable range: = $X_{theory} \pm 0.5 \times X_{theory}$ e.g. if the concentration measure by the organising laboratory is 40 all data outside the range of 20 - 60 will be rejected.
2b	Second Screening: statistical removal of outliers Tabulate remaining data (3rd column Table 1 "Outlier")	Procedure according to Rosner's test at a 95% confidence level. Rosner's test searches the data set for 1 up to a maximum of 10 outliers. See workbook on data analysis for documentation of Rosner's test.
2c	Calculate 95% Confidence Limit of data remaining after 2nd screening. Retain data values within LCL and UCL and tabulate (4th column Table 1 "95% CL")	Calculate upper and lower confidence limit of data remaining after 2nd screening: LCL = Mean – $1.96 \times SD/\sqrt{N}$ UCL = Mean + $1.96 \times SD/\sqrt{N}$ Retain data value (x) when: $LCL \leq x \leq UCL$
2d	Calculate accepted data range from residual data. Retain data values within LL and UL and tabulate (5th column Table 1 "Accepted")	Calculate: Reference Mean (X_{ref}) of remaining data, SD for (n-1) dof, and $SD_{(adjusted)} = SD^*\sqrt{2}$ Lower Limit (LL) = $X_{ref} - 3 \times SD_{(adjusted)}$ Upper Limit (UL) = $X_{ref} + 3 \times SD_{(adjusted)}$ Spread = $\pm 3 \times SD_{(adjusted)}$
2e	Verify if the calculated data spread is within the acceptable limits for the spread (Table B) If not acceptable, adapt the spread by correcting $SD_{(adjusted)}$	If $SD_{(adjusted)} > SD_{max}$ then: $SD_{(adjusted)} = Sd_{max}$ If $SD_{(adjusted)} < SD_{min}$ then: $SD_{(adjusted)} = Sd_{min}$ Recalculate LL and UL (from step 2d) with the updated $SD_{(adjusted)}$ and update column 5.
2f	Identify the original data values which are within the accepted range	Accept data value (x) when: Lower Limit (LL) $\leq x \leq$ Upper Limit (UL)
Repeat step 2 for all other samples-parameter combinations		
3	Create Table 2	Listing of Reference mean and accepted range (LL and UL) for all parameters in both samples

Step	Procedure	Comments
4	Create Table 3	For each laboratory, identify the parameters they have correctly analysed A laboratory must report **both** samples (A&B) within the acceptable range for the given parameter in order to have 'correctly' analysed the parameter.
5	Create Table 4	Compare result of current AQC round with previous round(s). Compare either with the previous round or with all previous rounds.
6	Construct Youden Plots per parameter	Parameter wise plot with results of all laboratories for sample A and B together with all data together with the values for LL and UL
(*) for each sample-parameter combination (2 x 9 = 18 numbers) 5 columns are presented in Table 0. For each sample-parameter combination the 5 columns contain: (1) raw data, (2) data after first screening (3) data after statistical outlier removal, (4) residual data and (5) accepted data.		