



Water and Wastewater PT Schemes Protocol

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Pasargad Quality Pioneers (PQP)
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History of Changes

No.	Date	Description of Change	Reviewed by	Revision number
1	August 01, 2015	First Edition	H.R. Dehnad	00
2	Oct 03 2016	Updating according to new PT specifications.	H.R. Dehnad	01
3	November 19, 2016	References to performance evaluation procedure and homogeneity and stability procedures are made. The subcontracted activities have been described.	H.R. Dehnad	02
4	December 25, 2016	Clause 4 about Evaluation of Homogeneity and stability has been added. Clause 5 subcontracted activities have been updated.	H.R. Dehnad	03
5	December 10, 2019	General changes to the contents of the protocol to cover the updated procedures in PQP PT process.	H.R. Dehnad	04
6	November 14, 2020	Clause 2-2 and 2-3, when the homogeneity is verified from the previous rounds, the homogeneity and stability are verified by using those data.	H.R. Dehnad	05

1- Organization of schemes

1-1- Planning

Planning of proficiency testing schemes in Pasargad Quality Pioneers (PQP) is done on an annual basis. The frequency of different PT schemes is planned based on the following factors:

- Requirements of regulatory bodies (e.g. national water and wastewater engineering organization)
- Feedbacks received from the participants
- Technical review of the organized PTs

The details of PQP PT programs including the technical specifications and the schedule of PT rounds are published in PQP annual PT calendar which is prepared and distributed to potential PT participants on an annual basis.

1-2- Registration in a proficiency testing round

At least two months prior to distribution of PT samples in a round, potential participants are informed about the technical specifications of the schemes (including the number of samples, analytes and their expected ranges, samples volume), the key dates of the round (including the registration deadline, sample distribution date, final report distribution date), registration fees and the terms and conditions of registration. The participants should submit their registration form to PQP in writing through email or fax. These information are specified in PT Registration form (FR-28) and PT program notification (FR-49).

The participants who registers in PQP proficiency testing rounds accepts the terms and conditions specified in General terms and conditions of PT participation (FR-50).

1-3- Confidentiality

In order to preserve the confidentiality of participants identity and results, each laboratory is randomly allocated a unique code number for the program to enable confidentiality of results. Reference to each laboratory in the final report is made by its code number.

National Water and Wastewater Engineering Company as a regulatory authority has requested PQP to directly provide the participants identity and results to them so the performance of the affected participants i.e. laboratories of water and wastewater companies are reported to National Water and Wastewater Engineering Company.

2- PT Samples

2-1- Sample preparation

PQP water and wastewater PT samples are a combination of real samples, synthesized samples, certified reference materials require dilution.

Details of test materials available are given in Specifications of PT programs list (LI-03). The test parameters are continually reviewed to ensure they meet the needs of current laboratory testing and regulatory requirements.

For some specific schemes, PQP subcontracts the sample preparation to competent subcontracts which are periodically evaluated to check if they meet the competency requirements.

2-2- Homogeneity of samples

The homogeneity of the samples is checked according to the procedures specified in ISO 13528 and IUPAC International Harmonized Protocol for the Proficiency Testing of Analytical Chemistry Laboratories according to Homogeneity and stability procedure (PR-15). Homogeneity testing is performed on a number of randomly selected samples and in order to confirm the homogeneity, between samples variation is compared against within samples variations or proficiency testing target standard deviation.



PQP subcontracts the homogeneity testing to competent subcontracts which are periodically evaluated to check if they meet the competency requirements.

For some specific schemes for which sufficient experimental data from the previous rounds are available to demonstrate that the samples are homogenous, the homogeneity is verified by using those data.

2-3- Stability of samples

The stability of the samples is checked according to the procedures specified in ISO 13528 and IUPAC International Harmonized Protocol for the Proficiency Testing of Analytical Chemistry Laboratories according to Homogeneity and stability procedure (PR-15). In order to evaluate stability, number of randomly selected samples are tested after an interval according to the specified deadline for the participants to submit their results. In order to confirm the stability, the difference between the samples results tested at different times is compared against the proficiency testing target standard deviation.

PQP subcontracts the stability testing to competent subcontracts which are periodically evaluated to check if they meet the competency requirements.

For some specific schemes for which sufficient experimental data from the previous rounds are available to demonstrate that the samples are homogenous, the stability is verified by using those data.

2-4- Packaging and transportation

PT samples have different containers which are specified in Specifications of PT programs list (LI-03). All of the samples have a standard label which includes the sample identification, safety symbols and the scheme name.

PT samples are transported using courier door-to-door posting services and should be received in 5 days.

3- Reporting of results

The participants should submit their results in PQP results submission online system (KPMD system) according to PT results registration in PQP software instruction (IN-04) and before the pre-specified deadline for submission of results. The participants who submit their results after the reporting deadline cannot be included in the final report. The final report is available to all participants regardless of whether their results were submitted or not.

Participants should use their test methods used in their laboratory for the analysis of their routine samples. Participants are asked to treat the PT samples as routine samples.

A statistical summary of different methods is provided for each analyte in the final reports to enable the performance of each method to be compared.

4- Data analysis and performance assessment

4-1- Setting assigned value

Information on the statistics used in these programs can be found in the final report. Methods for determining assigned values and the values for SDPA (standard deviation for proficiency assessment) used for individual samples are given in this scheme protocol. The details regarding calculation of SDPA are describe in Performance evaluation procedure PR-14.

The assigned value is the value selected as being the best estimate of the 'true value' for the parameter under test. The details regarding calculation of assigned value are describe in Performance evaluation procedure PR-14. The following approaches are used to calculate the assigned value for different parameters:

- **Formulation value**

Since the proficiency test samples were prepared by adding a specified proportion of different substances to distilled water so the assigned values have been derived by calculation from the masses of properties used.

- **Certified value**

Since the material used in this proficiency test is a certified reference material (CRM), its certified reference value is used as the assigned value X.

- **Consensus value**

The assigned value X for the test material used in this round of a proficiency testing scheme is the robust average of the results reported by all the participants in the round

4-2- Setting PT standard deviation

Standard Deviation of Proficiency Assessment (SDPA) is the measure of dispersion used in the evaluation of results of proficiency testing. The following approaches are used to calculate the SDPA for different parameters:

- **Prescribed value**

The standard deviation $\hat{\sigma}$ used to assess the proficiency of participants is calculated using fixed acceptance criteria specified in US NELAC¹ proficiency testing requirements and also reproducibility standard deviation values written in Standard Methods for Examination of Water and Wastewater.

- **Experience from previous rounds of a proficiency testing scheme**

The standard deviation is based on consensus statistics from previous rounds of the proficiency testing scheme.

- **Results from the proficiency testing scheme**

The standard deviation $\hat{\sigma}$ used to assess the proficiency of participants is derived from the results reported by the participants in the round

4-3- Uncertainty of the assigned value

The assigned value has an uncertainty that depends upon the method used to derive the assigned value.

When the assigned value is determined by formulation, the standard uncertainty is estimated by the combination of uncertainties of all sources of error, such as raw materials purity and gravimetric and volumetric measurements. When the assigned value is determined by the certified value, the reported uncertainty by the CRM producer is used as the assigned value uncertainty. When the assigned value is determined by consensus of participants' results, the estimated standard uncertainty of the assigned value can be calculated:

$$u = 1.25 \times s^* / \sqrt{p}$$

4-4- Calculation of performance scores (Z/ Z')

The performance of the participants are summarized using Z/ Z' scores. The Z-score is calculated using the following formula:

$$z = \frac{x - \mu}{SDPA}$$

In which:

¹ National Environmental Laboratory Accreditation Conference

x: participant result

μ : assigned value

SDPA: Standard deviation for proficiency assessment

If the measurement uncertainty of the assigned value (u_x) does not fulfill the following criteria:

$$u_x \leq 0.3 \times SDPA$$

Then the uncertainty of the assigned value is not negligible and should be considered in the interpretation of the results of the participants. For this purpose Z'-score is calculated as below:

$$Z' = \frac{x - \mu}{\sqrt{SDPA^2 + u_x^2}}$$

4-5- Interpretation of results

For the purposes of performance assessment for a single round, Z/ Z' scores are interpreted as follows:

|Z/ Z'| ≤ 2 Satisfactory Result

2 < |Z/ Z'| ≤ 3 Questionable result

|Z/ Z'| > 3 Unsatisfactory result

5- Information distributed to the participants

5-1- Final Reports

Final Reports are made available electronically in a period of approximately two months after the results submission deadline and can be downloaded from PQP online system (KPMD). This report is only accessible for the participants of the round and is not publicly available. The contents of reports may vary from scheme to scheme but include the requirements of ISO/IEC 17043 requirements. In case a participant requires a hard copy of the final report, it will be available to them upon request and acceptance of the additional expenses for such a purpose.

5-2- Individual Reports

The participants in a round of proficiency testing are provided with an individual report which summarizes their performance (Z/ Z' scores) for all of the analytes of the scheme which is available electronically and can be downloaded from PQP online system (KPMD).

The performance of late participants are only evaluated individually and they are provided with Summary Sheet (FR-32) in which their performance for each analyte is evaluated individually.

5-3- Certificates

All of the participants in a round of proficiency testing program are provided with a certificate to recognize their satisfactory performance or participation in the round of the PT program. If the participant's Z or Z' scores are less than or equal to |2| for all of the parameters in a scheme, the Certificate of Excellence is issued and if either one or more of the participant's Z or Z' scores are greater than |2|, the Certificate of Participation no. is issued. These certificates are available



electronically and can be downloaded from PQP online system (KPMD). In case a participant requires a hard copy of the certificate, it will be available to them upon request and acceptance of the additional expenses for such a purpose.

6- Complaints and appeals

The participants are able to submit their complaints through PQP quality assurance manager or in case of an appeal regarding the evaluation of their performance in a round, it can be submitted through the program coordinator. In one week period after receiving the complaint or appeal, PQP sends a letter to the participant to acknowledge the receipt of the customer/ appeal and ensure the participant that their complaint/ appeal will be handled in a timely manner.

The participant will be notified of the results after the actions are confirmed with a response letter.

7- Related Documents

- 7-1- PT Registration form (FR-28)
- 7-2- PT program notification (FR-49)
- 7-3- PT programs list (LI-03)
- 7-4- PQP software instruction (IN-04)
- 7-5- Performance evaluation procedure (PR-14)
- 7-6- Homogeneity and stability procedure (PR-15)
- 7-7- Summary Sheet (FR-32)