



Fera Science Ltd (Fera)

Protocol for Proficiency Testing Schemes

Version 6, April 2017

Part 5 – Fapas[®] Water and Environmental scheme
(LEAP)

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PREFACE

This Protocol is a series of inter-related documents. This document, Part 5, sets out specific details for the Fapas® Water and Environmental scheme (LEAP). Although this document duplicates some of the text in Part 1 – Common Principles, it cannot be used in isolation. Part 5 must always be read in conjunction with Part 1 and vice versa.

VERSION HISTORY

This Protocol was completely revised in 2009, superseding all proficiency testing scheme Protocols previously published by Fera in any of its incarnations.

Version 6 of April 2017, this version, supersedes Version 5 of September 2016. The changes are as follows;

Update organisation nomenclature throughout

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1. INTRODUCTION

1.1. Fera, PTG, Fapas® and LEAP

Fera was vested on 1 April 2015. Fera provides a wide range of proficiency testing (PT) schemes under the brand name of Fapas®. The management of these PT schemes is the sole task of one team within Fera, known internally as the Proficiency Testing Group (PTG).

For the purpose of this Protocol we use Fapas® to mean Fera PTG. Part 5 of this Protocol, i.e. this document, specifies details relating only to the Fapas® Water and Environmental scheme (LEAP).

Together with laboratory accreditation and the use of validated methods, PT is an important requirement of the EU Council Directive 98/83/EC [1] on the quality of water intended for human consumption. With the increasing demands for independent proof of competence from regulatory bodies and customers, proficiency testing is relevant to all laboratories testing water for quality and safety in every country.

1.2. Accreditation

Fera is a UKAS accredited Proficiency Testing Provider, No. 0009. Accreditation is conferred upon Fapas® Water and Environmental scheme (LEAP) in accordance with ISO/IEC 17043:2010 [2].

The formal schedule of the accreditation can be obtained from the United Kingdom Accreditation Service (UKAS) web site (Adobe PDF format) [3].

Unless otherwise specified in the detailed programme or brochure, all Fapas® Water and Environmental scheme (LEAP) PTs can be considered to be within scope of accreditation. The principal exceptions are the Emergency Scheme, and Taste and Odour Scheme. Details of the PTs and scope can be inferred from the published brochure and schedule of accreditation.

2. ORGANISATION OF WATER AND ENVIRONMENTAL SCHEME (LEAP)

2.1. Management System

The accredited management system covers all aspects of the PTs organised by Fapas® Water and Environmental scheme (LEAP), i.e. the same system applies whether a particular PT is within scope of accreditation or not.

The management system covers all aspects of the following PTs:

- Drinking Water Chemistry
- Environmental Waste Water, Surface Water, High Salinity Water and Soil
- Drinking Water Microbiology, Parasitology and Environmental Legionella
- Emergency, and Taste and Odour

Note that Chemistry PTs are grouped according to their standard analysis.

3. PARTICIPATION IN SCHEMES

3.1. Test Material Preparation and Homogeneity

Fapas® Water and Environmental scheme (LEAP) test materials are a combination of real samples, standard solutions and standard concentrates that require dilution. The test material preparation is

carried out by subcontracting laboratories. Liquid samples, by their nature have a high degree of natural homogeneity and this, coupled with rigorous process control during preparation, means that homogeneity testing is not normally required.

3.2. Dispatch and Receipt of Test Materials

All Fapas® Water and Environmental scheme (LEAP) test materials are sent by courier and so can be tracked on-line throughout their journey. It is the responsibility of the customer to anticipate an email on the day of dispatch advising them of the tracking number and then to monitor the progress of their samples. It is particularly important for non-UK participants to track their samples to ensure a smooth transit through their country's Customs. Fapas® cannot be held responsible for delays arising at Customs.

3.3. Analysis of Test Materials

Many Fapas® Water and Environmental scheme (LEAP) test materials are supplied in the form of a water sample and an associated spiking concentrate. Full details of how to add the spiking solution to the water sample are supplied in the instructions (provided electronically via email or downloaded from the Fapas® website, www.fapas.com). It is the responsibility of participants to read these instructions and follow them exactly prior to conducting the actual analysis of the test material. Fapas® cannot be held responsible for any problems arising from failure to comply with these instructions.

An indication of the likely concentration range(s) in the test material will only be given if the test material is in the form of a standard solution or concentrate that must be diluted prior to analysis.

Example instructions are available on request from Fapas®.

3.4. Follow-Up Services

Fapas® does not sell surplus test materials from the batches used for Fapas® Water and Environmental scheme (LEAP) PTs.

Most Fapas® Water and Environmental scheme (LEAP) Reports produced since 2003 are available for purchase. Prices are available on request. Participants in all the Fapas® schemes have free access to an electronic copy of reports for those tests for which they have registered. Electronic copies of reports are available on request and a charge will be made for these.

If a participant wishes to obtain advice on any aspect of their performance they should contact Fapas® by email (info@fapas.com) in the first instance. Participants must note that Fapas® may offer assistance in the form of a broker service whereby Fapas® will either anonymously or, subsequent to all parties agreeing to waive their confidentiality, pass on the participant's inquiry to an expert laboratory/external advisor.

4. PERFORMANCE ASSESSMENT

The Fapas® Water and Environmental scheme (LEAP) PTs express participants' results as z-scores, except for parasitology PTs where the performance assessment takes the form of a general comparison of participants' results and percentage recovery. The Emergency and Taste and Odour PTs are not performance assessed (details below).

The standard deviations for each PT are derived as detailed below.

4.1. Drinking Water Chemistry PT

4.1.1 Inorganic Groups

For Chemistry PTs, z-scores are used for quantitative results.

Regulation 21(2)(d)(iii) [4] outlines the performance required of analytical systems and the maximum tolerable errors are specified in the following way:

- The maximum tolerable total error of individual results should not exceed C or 20% of the result, whichever is the greater. Each analytical programme has its own analytical range requirement. At the lower end of such a range, knowledge of the actual concentration provides no additional benefit. This concentration, known as the Lower Level of Interest (LLI) equates to C, where C = one tenth of the Prescribed Concentration Value (PCV) [5].

The reason for this is that, as concentration decreases, the maximum tolerable error of 20% becomes more difficult to achieve. At low concentrations the satisfactory range will appear small and is not practical, thus, C is used.

Some measurands will have lower levels of interest (LLI) *not* set at 1/10th of the PCV, but set by perception, based on experience of the quality of data that is achievable by laboratories.

Satisfactory Range Calculation

If robust mean, median or theory value is less than (<) lower level of interest (LLI) then upper and lower range is set as follows:

$$\text{Upper} = +2 \times \text{LLI}$$

$$\text{Lower} = 1\% \text{ of LLI}$$

If robust mean, median or theory value is greater than (>) lower level of interest then use one of following:

$$\pm 20\% \text{ or } \pm \text{LLI} \text{ whichever is the greater}$$

Standard Deviation (σ_p)

From the satisfactory range one of two options is used:

- (i) If assigned value is (<) LLI then $\sigma_p = (\text{Upper} - \text{Lower})/4$
- (ii) If assigned value is (>) LLI then $\sigma_p = \pm 20\%$ of assigned value divided by 2 or LLI divided by 2 whichever was used to set satisfactory range.

pH is a special case where σ_p is set at ± 0.2 units. [5]

Uncertainty of assigned value

Where the assigned value has been derived from the consensus of participants' results, the uncertainty is also derived from the participants' results (see the Protocol Part 1 – Common Principles).

Where the assigned value has been derived from the theoretical spike value (defined by Fapas[®]), the combined uncertainty of the spiked sample preparation has been calculated by Fapas[®] Water and Environmental scheme (LEAP) to be no more than about one-tenth (10%) of σ_p . This value is negligible in relation to performance assessment in Fapas[®] Water and Environmental scheme (LEAP).

Where the assigned value has been taken as a certified reference value, the uncertainty is expressed as the expanded uncertainty $U(U_{CRM})$ taken from the reference material's certificate.

4.1.2 Organics

The standard deviation for proficiency, σ_p , for Drinking Water organic groups is set with reference to performance requirements in Water Regulations [5], at a value that reflects best practice for the analyses in question.

The appropriate tolerable error, for each analyte, is dependent on the assigned value. If the assigned value is greater than or equal to (\geq) 50% of the PCV [5] then σ_p is set at 12.5% of the assigned value. If the assigned value is less than (<) 50% of the PCV, σ_p is set at 6.25% of the PCV. Where no PCV is available, 0.1 $\mu\text{g/L}$ is used as the PCV.

The uncertainty of the assigned value is assessed in the same way as for the inorganics (section 4.1.1 above).

4.2. Environmental Waste Water, Surface Water, High Salinity Water & Soil Chemistry PTs

The standard deviation for proficiency in environmental matrix PTs is 10% of the assigned value, except pH where it is 0.1 pH units.

4.3. Drinking Water Microbiology/Parasitology & Environmental Legionella PTs

Test materials

Test materials for Drinking Water Microbiology and Environmental Legionella PTs are usually in the form of a tablet, provided with or without an associated diluent. Test materials for identification PTs (option in Drinking Water Microbiology) are an agar slope containing the organism. Test materials for Parasitology PTs are tap water concentrates in phosphate buffered saline (PBS) and concentrated suspensions in PBS, containing a combination of *Cryptosporidium* oocysts and *Giardia* cysts. Instructions are provided for sample preparation prior to analysis.

Assessments

Qualitative assessments for Drinking Water Microbiology and Environmental Legionella PTs report results as either Satisfactory or Not Satisfactory, against the identification of the verified organism present in the test material. The assessment will be based only on the reported genus. Abbreviations are not acceptable due to the possibility of their misinterpretation.

Quantitative assessments for Drinking Water Microbiology and Environmental Legionella PTs are by z-score.

Parasitology PTs are not assessed by z-score but evaluated by a general comparison of participants' results. A mean and standard deviation is calculated for each laboratory, in addition to the overall robust mean and standard deviation for all results. The efficiency of recovery of *Cryptosporidium* oocysts and *Giardia* cysts is assessed using the following formula:

$$\text{Efficiency} = \frac{\text{observed number of Oocysts or Cysts}}{\text{known initial inoculum}} \times 100$$

An efficiency recovery value of 30% or greater is considered satisfactory.

Data Analysis and Standard Deviation (σ_p)

The standard deviation for proficiency in quantitative assessments for Drinking Water Microbiology PTs used to be either the robust standard deviation (where the robust mean is set as the assigned value) or the standard median absolute deviation sMAD (where the median is set as the assigned value). The use of an internal standard deviation has several disadvantages because it is not independently-derived.

A new data assessment process has now been implemented which does two things. Firstly, it transforms the raw data by square root. Secondly, it applies an externally-derived fixed σ_p which is organism-dependent.

The square root transformation was derived from looking at several years' worth of data and applying different transformation methods, including \log_{10} , \log_{1n} and square root. Although log transformations work well for very high counts in food matrices, they are less appropriate for the observed distributions and counts in water microbiology. Square root transformation, however, provides quasi-normal, symmetrical datasets. When combined with a robust method of location, the assigned value can be reliably determined from the consensus.

The standard deviation for proficiency assessment (σ_p) was derived from analysing the same historic Fapas[®] Drinking Water Microbiology PTs. The values for σ_p used are 1.5 SQRTcfu/100 ml for Total

Coliforms, *Escherichia coli*, *Pseudomonas aeruginosa* and *Clostridium perfringens* and 1.25 SQRTcfu/100 ml for Enterococci and 1.25 SQRTcfu/ml for Colony Counts.

This data analysis was verified in parallel on the results submitted for three distributions in October-December 2015 (MICRO 93, MICRO 94 and MICRO 95). All subsequent Fapas® Drinking Water Microbiology PTs reports from January 2016 contain z-scores calculated only with the new square root transformation and fixed standard deviation for proficiency.

This change does not apply to the Legionella proficiency tests. The standard deviation for proficiency in quantitative assessments for Drinking Water Legionella PTs is set at 0.55 log₁₀ cfu/L. This was updated from the previous 0.35 log₁₀ cfu/L following discussion and agreement at the Fapas® Water and Environmental scheme (LEAP) Advisory Committee meeting of 24 September 2013.

This method of microbiology data analysis is approved in ISO/TS 22117:2010 [6].

Interpretation of assessments

This fixed standard deviation has been set at a value that reflects best practice for the analyses, and it is used in conjunction with the assigned value to calculate the z-score. The results received from participants (cfu/100 ml or cfu/ml) are square root transformed by Fapas® and the new fixed standard deviation for proficiency assessment applied. Participants are still required to submit results in original units of cfu/100 ml or cfu/ml.

As a result of this development participants will be able to observe trends over a period of time. This will help identify any ongoing problems and improve assessments of participants' performance. The consideration of a set or sequence of z-scores over time provides more useful information than a single z-score.

4.4. Emergency Scheme and Taste and Odour Scheme

The Emergency Scheme and Taste and Odour Scheme replicate unforeseen events occurring at drinking water supplies. Both of these schemes are simulated contamination events carried out in real time and participants have to react and report the results as if it was a real incident.

The Emergency Scheme is for drinking water companies to respond to a toxic chemical contamination incident. Participants register for the PT which is scheduled twice per year with only the month of distribution known in advance. The participants only know that the PT has started when they receive the samples, together with a scenario of the incident. Results of analyses are reported together with expert commentary from the participants on the seriousness of the contamination. Receipt of results at Fapas® are time-logged.

The Taste and Odour Scheme is for drinking water companies to respond to consumer complaints of unusual taste or odour in the water supply. The PT sample is contaminated with chemicals that would give an unusual taste or odour. The participants are required to identify the contaminants using analytical chemistry techniques but not using taste organoleptic methods. A scenario of the incident is provided with the sample and results of analyses are reported together with expert commentary from the participants on the possible sources of the contaminants.

For the Emergency Scheme, intended results are distributed to participants one day after the close of the PT and before the formal PT report is issued.

Reports of the PTs detail all of the participants' results, expert commentary and brief method details. As well as identification, participants can submit quantitative data. No performance assessments are carried out for these PTs. Instead, an open meeting is held for all the participants of the different PTs once per year. The meeting provides a discussion forum at which best practice on identification methods is discussed. The meeting is held at Fera under the Chatham House Rule to enable free discussion only within the confines of the meeting.

5. REFERENCES

- 1 Council Directive 98/83/EC of 3 November 1998 on the quality of water intended for human consumption (European Drinking Water Directive).
- 2 ISO/IEC 17043:2010, Conformity assessment – General requirements for proficiency testing.
- 3 <http://www.ukas.com>, accessed 17/03/2017
- 4 Guidance on safeguarding the Quality of Public Water Supplies 1989.
- 5 The Water Supply (Water Quality) Regulations 2016 No. 614, Water, England and Wales
- 6 ISO/TS 22117:2010, Microbiology of food and animal feeding stuffs – Specific requirements and guidance for proficiency testing by interlaboratory comparison.

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