



The Food and Environment Research Agency

Protocol for Proficiency Testing Schemes

Version 2, January 2012

Part 5 – LEAP[®] Scheme

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PREFACE

This Protocol is a series of inter-related documents. This document, Part 5, sets out specific details for the Laboratory Environmental Analysis Proficiency (LEAP[®]) Scheme. 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 the Food and Environment Research Agency (previously, Central Science Laboratory), i.e. all previous editions of the separate FAPAS and FEPAS Protocols.

Version 2 of January 2012, this version, supersedes Version 1 of November 2009. The changes are as follows;

Change from LEAP[™] to LEAP[®]

- 1.1 Addition of text taken from Reports on the rationale for water quality PT
- 1.2 Amendments to reflect publication of standard ISO/IEC 17043 and compliance with its requirements
- 3.3 Electronic instructions, not covering letters
- 4.1.2 Standard deviation update
- 4.3 Update on qualitative microbiology assessments
- 5 References updated

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1. INTRODUCTION

1.1. Fera, Defra, PTG and FAPAS

The Food and Environment Research Agency (Fera) is an Executive Agency of the UK Government Department for Environment, Food and Rural Affairs (Defra). Fera provides a wide range of proficiency testing (PT) schemes.

The management of these PT schemes is the sole task of one of many teams within Fera. Known internally at Fera as the Proficiency Testing Group (PTG), this team provides Fera's PT schemes globally under the brand name of FAPAS PT. The Laboratory Environmental Analysis Proficiency (LEAP[®]) scheme is one of these PT schemes.

For the purpose of this Protocol we use FAPAS PT to mean Fera PTG.

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

LEAP[®] Scheme is accredited by the United Kingdom Accreditation Service (UKAS). Accreditation is conferred upon LEAP[®] Scheme 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].

2. ORGANISATION OF LEAP[®] SCHEME

2.1. Management System

The scope of the LEAP[®] Scheme's accreditation covers all aspects of the following PTs organised by LEAP[®] Scheme:

- Potable Water Chemistry
- Effluent, Waste Water, Contaminated Land and Hazardous Waste
- Potable Water Microbiology/Parasitology

Note that Chemistry PTs are grouped according to their standard analytical methodology.

3. PARTICIPATION IN SCHEMES

3.1. Test Material Preparation and Homogeneity

LEAP[®] Scheme test materials are a combination of real samples, standard solutions and standard concentrates that require dilution. 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 LEAP[®] Scheme 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 PT cannot be held responsible for delays arising at Customs.

3.3. Analysis of Test Materials

Many LEAP[®] Scheme 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 PT 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. LEAP[®] Scheme 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 LEAP[®] Scheme.

3.4. Submission of Results and Outline Methodology

The general results submission requirements are detailed in Part 1 of this Protocol.

3.4.1 Rapid Indication of Consensus of Results

During the period from approximately 5 days after the closing date until the publication of the report participants can access, via FAPAS SecureWeb, a table listing the median values of the submitted results for each analyte in the round. These median values are only rapid indicators of the consensus of submitted results. They are NOT the assigned values. They are indicative only and for **use at participants' own risk**. Fera accepts no liability in any circumstances for the use/misuse of these values.

3.5. Follow-Up Services

FAPAS PT does not sell surplus test materials from the batches used for LEAP[®] Scheme PTs.

Most LEAP[®] Reports produced since 2003 are available for purchase. Prices are available on request. Participants in all the FAPAS PT 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 PT by email (info@fapas.com) in the first instance. Participants must note that LEAP[®] Scheme may offer assistance in the form of a broker service whereby LEAP[®] Scheme 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 LEAP[®] Scheme PTs express participants' results as z-scores, except for parasitology PTs where the performance assessment takes the form of percentage recovery.

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

4.1. Potable Water Chemistry PT

4.1.1 Inorganic Groups

For Chemistry PTs (single determinations), 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: -

Upper = +2 x LLI

Lower = 1% of LLI

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

+/- 20% or +/- LLI 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 $\sigma_p = +/-20\%$ of assigned value divided by two 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]

Potable Water Chemistry PTs may also include multiple determinations, in which participants report replicate results. Each laboratory will then have a bias, calculated as:

bias = laboratory mean unspiked value - robust mean of all laboratories unspiked value

The bias target is calculated as:

LLI/2 or 10% of overall mean whichever is the greater

The precision (standard deviation) target is calculated as:

LLI/4 or 5% of overall mean whichever is the greater

An additional assessment in the Chemistry PTs is the Spiked Sample Recovery. An individual laboratory mean value for the unspiked sample replicates is calculated along with an individual mean value for the spiked sample replicates. The recovery for each laboratory is then calculated as follows:

$$\text{recovery} = \text{mean spiked value} - \text{mean unspiked value}$$

$$\% \text{ recovery} = (\text{mean spiked value} - \text{mean unspiked value}) / (\text{nominal spike level} \times 100)$$

The recovery target is calculated as +/- 10% of nominal spike level or +/- LLI/4, whichever is the greater.

4.1.2 Organics

The standard deviation for proficiency, σ_p , for potable 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.

4.2. Effluent, Waste Water, Contaminated Land & Hazardous Waste PTs

The standard deviation for proficiency in Effluent, Waste Water, Contaminated Land & Hazardous Waste PTs is 10% of the assigned value, except pH where it is 0.1 pH units.

4.3. Potable Water Microbiology/Parasitology PTs

Qualitative assessments for Potable Water Microbiology 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.

The standard deviation for proficiency in quantitative assessments for Potable Water Microbiology PTs will 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 standard deviation for proficiency in quantitative assessments for Potable Water Legionella PTs is set at 0.35 \log_{10} cfu/L.

The Potable Water Parasitology PTs differ in that the efficiency of recovery of *Cryptosporidium* oocysts and *Giardia* cysts is assessed, as well as reporting the enumeration. These PTs do not issue z-scores but instead there is 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.

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/about-accreditation/accredited-bodies/proficiency-testing-organisations.asp>
- 4 Guidance on safeguarding the Quality of Public Water Supplies 1989.
- 5 Guidance on the implementation of the Water Supply (Water Quality) Regulations 2000 (as amended) in England and the Water Supply (Water Quality) Regulations 2010 in Wales (Published September 2010).

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