



The Food and Environment Research Agency

Protocol for Proficiency Testing Schemes

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Part 1 – Common Principles

PREFACE

This Protocol is a series of inter-related documents. Part 1, this document, sets out an overview of, and the principles common to, all of the PT schemes provided by the Food and Environment Research Agency. Subsequent parts give scheme specific details. It follows that neither Part 1, nor any of the other parts, can be used in isolation. Part 1 must always be read in conjunction with a scheme specific supporting part and vice versa.

This document, together with its supporting parts, supersedes **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.

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1. INTRODUCTION

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®.

FAPAS® is an acronym for Food Analysis Performance Assessment Scheme. The other branded PT schemes run by PTG are the Food Examination Performance Assessment Scheme (FEPAS®), Laboratory Environmental Analysis Proficiency scheme (LEAP™), Genetically Modified Materials Analysis performance scheme (GeMMA) and plant health diagnostics (PhytoPAS). This Protocol, Part 1, should be read in conjunction with the scheme-specific parts. For FAPAS® in its entirety, see also Part 2 of the Protocol. For FEPAS®, see also Part 3. For GeMMA scheme, see also Part 4. For LEAP™ scheme, see also Part 5. PhytoPAS is still in its infancy, hence, there is no independent Protocol part for PhytoPAS yet.

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

1.1. What is PT?

ISO/IEC 43-1: 1997 [1] defines PT as the use of inter-laboratory comparisons to determine the performance of individual laboratories for specific tests or measurements and to monitor a laboratory's continuing performance.

The demand for independent demonstration of competence, from regulatory bodies and customers, means that proficiency testing is relevant to all laboratories testing samples for quality and safety. Hence, it is a requirement of accreditation to ISO/IEC 17025 [2] that the laboratory takes part in a PT scheme, if a suitable scheme exists. In particular, for laboratories entrusted with the official control of food and feeds, Article 12 of EU Regulation (EC) 882/2004 [3] requires such laboratories to be assessed and accredited in accordance with ISO/IEC 17025. Proficiency testing is therefore a legal requirement for these laboratories. Thus, together with the use of validated methods and internal quality control, proficiency testing is an essential element of laboratory quality assurance.

In summary, PT is a way of checking the accuracy [4] of results from laboratories.

1.2. Accreditation and PT

Accreditation is a completely separate concept to PT. Accreditation requires the formal, external, assessment of an organisation's documented procedures against a relevant International Standard.

The relevant conformity standard for laboratories in the field of testing is ISO/IEC 17025: 2005. Compliance with ISO/IEC 17025 alone cannot guarantee that the procedures give accurate results. Only the external check of a proficiency test can confirm the results are accurate – hence the requirement within ISO/IEC 17025 for laboratories to take part in PT schemes.

As yet there is no full ISO standard detailing the requirements for the accreditation of PT providers, only a guide, ISO/IEC 43-1: 1997. A full ISO standard, to be known as ISO 17043, is being developed. The starting text for this proposed standard was taken from ILAC G13: 2007 [5]. This is a document that expands upon ISO Guide 43 and cross-refers to relevant sections of ISO/IEC 17025 and ISO 9001. At present the United Kingdom Accreditation Service (UKAS) confers accreditation on PT Providers in accordance with ISO Guide 43 through assessment against ILAC G13: 2007.

It must be stressed that taking part in a PT scheme does **NOT** confer accreditation upon a laboratory. This applies even if the PT provider is, as is FAPAS®, accredited for the provision of PT schemes.

2. ORGANISATION OF SCHEMES

2.1. Administration

All PT schemes provided by FAPAS[®] are administered in keeping with internationally agreed principles. In particular those set out within the IUPAC International Harmonized Protocol for the Proficiency Testing of Analytical Chemistry Laboratories [6]. The original (1994) version of this International Harmonized Protocol was derived from the original (1991) FAPAS[®] Protocol while the recent revision (2006) drew heavily upon the experience of FAPAS[®] in delivering PTs in the intervening years.

Each PT scheme has its own Advisory Committee, which meets at least annually. The Advisory Committees comment upon the relevant programme of PTs planned by FAPAS[®] for the forthcoming year and discuss any scientific issues arising from PTs conducted in the current year. Committee members are available to advise FAPAS[®] staff at any point during the year and group email correspondence is frequently used to facilitate discussions. A list of current Advisory Committee members and the terms of reference are available on request from FAPAS[®].

The day to day running of an individual PT is the responsibility of a 'Round Administrator', i.e. a designated member of staff, overseen by a 'Scheme Co-ordinator'. Ultimate responsibility for all FAPAS[®] PTs lies with the Head of Group. Expert advice to support all staff in these duties is readily available from within Fera and from a variety of external sources. External advisors are selected on the basis of their personal expertise and not their affiliation; they need not be members of the relevant Advisory Committee. When consulting experts, FAPAS[®] will not disclose any participant information, purely scientific information will be exchanged, see below.

2.2. Confidentiality

All information held by FAPAS[®] about participants, including their z-scores, is confidential and will not be disclosed to anyone unless explicitly agreed by the participant for a particular purpose. To preserve this confidentiality participants receive reports giving all the results for that PT but without identifying individual laboratories. The laboratory code numbers used in reports are assigned in order of receipt of results from participants. Participants will be assigned the same code number in different PTs only by chance.

To avoid any conflict of interest / breach of confidentiality, if any of the various analytical testing teams elsewhere within Fera wished to participate in a PT they would be treated in exactly the same manner as any other participant. They would not have access to details of any other participants. Likewise, when FAPAS[®] seeks expert advice from other parts of Fera (or indeed any external source) it will not disclose any information that would breach participant confidentiality.

Once reports are issued to participants they are regarded as being in the public domain but all PT reports issued by FAPAS[®] are UK Crown Copyright, which cannot be assigned to other publishers. Anyone wishing to use data from within FAPAS[®] reports for their own publications should first seek permission from FAPAS[®]. It should be noted that this request for respect of copyright cannot preclude publications exploiting FAPAS[®] data being distributed without the prior knowledge or approval of FAPAS[®].

2.3. Typical Timetable

FAPAS[®] provides on-going PT schemes, where test materials are distributed on a regular basis every year as well as so-called 'closed' PT schemes, where the test materials are distributed at the time and request of a commissioning client.

For ease of planning and timetabling FAPAS[®] advertise their on-going schemes in annual blocks, from 1 April to 31 March the following year. These annual programmes of proficiency tests are compiled by FAPAS[®] in conjunction with the Advisory Committee for each Scheme. They are generally

published in December in anticipation of the following April-March. Where short date formats are published, the UK convention of DD/MM/YY is employed.

The outline process of conducting a single proficiency test is as follows:

- a) Preparation of test materials, including homogeneity testing.
- b) Dispatch of test materials on advertised date from FAPAS[®], York, UK.
- c) Participants analyse test materials and report results by a given date. Generally the closing date is six to eight weeks from the dispatch date though for certain analyses where the analyte/matrix combination potentially are unstable a much shorter time scale may be set.
- d) Results subjected to statistical analysis by FAPAS[®].
- e) Distribution of final report to all participants. Generally the report is issued within a month of the PT closing date but FAPAS[®] reserves the right to extend this period in cases where the statistical evaluation proves to be atypical.

Participants will be kept informed by email if a delay arises at any of these stages.

2.4. Management System

The quality management system for the whole of Fera is certified to ISO 9001 [7]. In addition, the majority of the work of FAPAS[®] is accredited by UKAS. The formal accreditation certificate is available on the FAPAS[®] web site [8] (Adobe PDF format), while the current formal schedule detailing the scope of this accreditation can be obtained from the United Kingdom Accreditation Service (UKAS) web site (Adobe PDF format) [9].

The scheme specific supporting parts to this document reiterate the accreditation status of each PT scheme.

2.5. Subcontractors

FAPAS[®] does not have any laboratory facilities of its own. Test material preparation and homogeneity testing is carried out by subcontractors. Homogeneity testing may be carried out by a different laboratory to the one that prepares the test material. FAPAS[®] maintains a list of approved subcontracting laboratories and regularly reviews the service received. Where possible, FAPAS[®] will only use subcontracting laboratories who hold accreditation to recognised international standards (ISO/IEC 17025 [2], for example). Subcontracting laboratories may also participate in FAPAS[®] PTs. In this situation, the subcontracting laboratory participation will be treated in exactly the same way as all the other participants, and the same rules of confidentiality will apply.

3. PARTICIPATION IN SCHEMES

None of FAPAS[®] PT schemes stipulate a minimum number of level/rate of participation. Participants do not necessarily have to analyse for all the analytes in a test.

3.1. Enrolment and Fees

The programme of all our PTs is available on our web site, www.fapas.com. Customers are encouraged to place their orders on-line by browsing these programmes and compiling a 'wish list'. If the customer is a previous participant and has access to the secure pages of our web site they can convert their 'wish list' into a formal order on-line. New customers can either use the 'wish list' to request a quote or print it out and fax it to us. Alternatively, hardcopies and/or PDF files of the programmes are available from FAPAS[®], at the address shown on the final page of this document.

PT order confirmations are automatically emailed to customers on completion of the ordering process. The confirmation email contains a link to a printer friendly version of the order, held within the

customer's secure pages on our web site. Where such electronic communication is not possible, a hardcopy will be posted. It is the responsibility of the customer to check that FAPAS[®] has processed their requests correctly, i.e. that they are enrolled in the correct PTs.

Fees etc. are fully detailed both on-line and in the hardcopy programmes. FAPAS[®] reserves the right to withhold test materials and/or PT reports from participants if payment is delayed.

Formal standard terms and conditions for proficiency testing schemes are available with the programmes or separately, either from the address given at the end of this document or from our web site (PDF format) [10].

3.2. Agents

Agents are appointed by FAPAS[®] in some countries. The advantages to participants of using the agent are to register locally to participate in FAPAS[®] PTs and the facility to pay in local currency. Agents will also liaise with FAPAS[®] on the participant's behalf for any queries or problems. Agents can also help samples pass more easily through customs. Details of participants' performance in the PTs are not disclosed to the agents. The list of agents is available from the website, www.fapas.com.

3.3. Dispatch and Receipt of Test Materials

All test materials are distributed with an associated covering letter. This letter gives all the necessary details about storage on receipt, type of analysis required, and reporting of results, including the required units, etc.

It is the responsibility of participants to read these instructions and follow them. FAPAS[®] cannot be held responsible for any problems arising from failure to comply with these directions.

It is the responsibility of the participant to contact FAPAS[®] if they have not received the test material within agreed timescales, as set out in the Fera Standard Terms and Conditions for Proficiency Testing Schemes.

Delays to the dispatch of test materials occasionally arise, typically when the prepared test material fails to reach the required level of homogeneity or stability. If the dispatch of a test material has to be delayed for any reason, then participants will be notified of this fact by email or fax prior to the advertised dispatch date. FAPAS[®] cannot be held responsible if participants overlook this notice of delay.

3.4. Analysis of Test Materials

If the PT is to yield maximum benefit as an external check on the routine working of participants' methods then the sample should not be given any special treatment. Hence, participants are free to use whatever method of analysis they wish. On the occasions where the method is known to be empirical (i.e. the result is dependent on technique) participants are still free to use whatever method they wish. In order to obtain a comparable set of results for statistical assessment, however, FAPAS[®] may advise participants that only the results submitted for a given method will be used to derive an assigned value by consensus.

3.5. Submission of Results and Outline Methodology

The reporting of results within the requested time scale and in the specified units is part of the performance assessment.

Participants are requested to submit their results and methods via the secure pages on our web site. The unique UserID and Password required to access these pages is given in the paperwork that accompanies each test material dispatched. While the submission of a result is a prerequisite for a performance assessment, participants are not obliged to submit their methodology.

If participants have no Internet access then FAPAS® may arrange to accept their results (but not their method details) by fax or post – providing the data are submitted on the correct form, which will have been sent with the test material *and* it reaches us before the closing date. A charge may be made for fax or postal submission.

Acceptance, or otherwise, of results submitted after the closing date is at the discretion of the Round Administrator.

3.5.1. Collusion and Falsification of Results

Collusion, either between participants or between individual participants and the scheme provider, is contrary to professional scientific conduct. It serves only to nullify the benefits of proficiency testing to customers, accreditation bodies, and analysts alike. Collusion is, therefore, to be strongly discouraged.

As a preventative measure FAPAS® reserves the right to distribute more than one test material within a PT so that participants cannot compare results directly. Ultimately though it is the responsibility of the participating laboratories to avoid collusion or falsification of results. Laboratories found to be falsifying results may be refused participation in subsequent proficiency tests.

3.6. Report Distribution

Participants are advised in the letter that accompanies the test material when to expect the publication of the report. FAPAS® aims to do this as soon as is practical after the closing date of the PT. Participants should note that our quality procedures involve extensive cross-checking and scrutiny by several people under the guidance of the Test Administrator and consequently this means the process takes anywhere between 3-8 weeks depending on the complexity of the data.

All reports are distributed in Adobe PDF format. They are both password secured and digitally signed to ensure that they cannot be altered in any way. The digital signature automatically validates when the PDF file is opened using Adobe Reader v7 or higher on a PC with access to the Internet. Reports are only available for download to the named contact(s) for the PT in question. An example FAPAS® report is freely available for download from the website, www.fapas.com.

Printed and bound copies of the reports are available at extra cost. Participants should note that this medium is inherently less secure than the digitally signed electronic PDF files.

3.7. Follow-Up Services

If a participant wishes to obtain advice on any aspect of their performance they should contact FAPAS® by email in the first instance. Participants must note that FAPAS® is most likely to 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.

Surplus test materials from the batch used for the PT may be available for purchase. These samples are not Certified Reference Materials. However, reference materials for the food analysis sector are not numerous and surplus FAPAS® test materials may be the only source of a suitable quality control material.

Outline details on the availability or otherwise of such quality control materials are given in the relevant report. The exact stock level of any given quality control material can be checked via our web site.

4. PERFORMANCE ASSESSMENT

The statistical model used by FAPAS® is set out fully within the International Harmonized Protocol [6]. In summary, as indicated in the Introduction, the purpose of a FAPAS® PT is to check the accuracy of

results submitted by the participating laboratories. This check is achieved typically by comparing participants' results to some estimate of the 'true' value.

If the results submitted are **quantitative** then this comparison will be in the form of a numerical score. Semi-quantitative (< or >) data are not assessed, *except* where detailed in the relevant scheme specific supporting parts of this Protocol.

The comparison for **qualitative** results will be against the answer anticipated by formulation, taking account of the consensus of participants' results.

The results submitted to a single PT represent the final product in a complex string of actions carried out by the participants, from sample receipt to results reporting. As such they encompass all aspects of a laboratory's performance. A mistake, however trivial, at any stage will contribute to the final outcome.

It is unwise to view any performance assessment as anything other than a snapshot of the whole laboratory performance at the time of the PT.

4.1. Scoring

4.1.1. Why score?

The advantages of expressing participants' results as a *standardised* score are that:

- they are simple and transparent,
- they present participants' results in a readily understood form,
- they permit comparison over time,
- when tabulated and charted, they place individual performance in the overall context of the PT.

When the *standardised* score incorporates a prescribed value that represents limits of acceptable variation for the analysis in question then the score embodies the concept of fitness-for-purpose, i.e. the balance between expending considerable time and effort (= expense) on an analysis to get a highly accurate result vs. carrying out a rough and ready procedure that only provides an indication of the level present and so be of limited use/require further analysis.

4.1.2. Types of scores

A variety of standardised scores is available. This Protocol presents only two such scores but this does not preclude FAPAS[®] from adopting alternatives, if so advised by our statistical experts.

4.1.2.1. z-Scores

FAPAS[®] favours the use of z-scores for the reasons given above. Notably because when the standard deviation is based on a fit-for-purpose criterion, i.e. it is a prescribed 'standard deviation for proficiency', then the significance of the performance assessment is immediately apparent, no matter what the concentration or identity of the analyte, the nature of the test material or the physical principle underlying the analytical measurement.

A z-score combines an estimate of the error of a result with a standard deviation:

$$z = \frac{(x - x_a)}{\sigma_p}$$

where x = the result reported by the participant

x_a = the assigned value

and σ_p = the standard deviation for proficiency

The derivation of the assigned value and the choice of fit-for-purpose standard deviations for proficiency are more complex.

In all FAPAS[®] PTs, the 'assigned value' is the best estimate available to FAPAS[®] of the 'true' value. The assigned value can be set as a:

- consensus value
- formulation level
- certified reference value

Suitable algorithms for the derivation of a consensus value are readily available [11, 12, 13]. The assigned value is almost invariably taken by FAPAS[®] to be the consensus value.

Fit-for-purpose standard deviations for proficiency (informally, the 'target value') can be obtained from:

- predictive models, e.g. modified Horwitz Equation [14]
- collaborative trials / method performance studies
- legal definition
- expert opinion

The Horwitz function, describing the trend of standard deviation of reproducibility found in collaborative trials, represents fitness-for-purpose in the food sector over a wide range of concentrations. It is therefore used by FAPAS[®] in the majority of instances.

The report for each PT will give full details on the choice and calculation of both the assigned value and the standard deviation for proficiency assessment.

4.1.2.2. Q-scores

On very rare occasions, where FAPAS[®] is unable to set an appropriate standard deviation for proficiency assessment, it may be appropriate to calculate a 'Q-score':

$$Q = \frac{(x - x_a)}{x_a}$$

where x = the result reported by the laboratory
 x_a = the best estimate of the 'true' value

This type of score only indicates the relative error of a result.

4.2. Interpreting Scores

4.2.1. Interpreting z-Scores

The guiding principle of scoring in FAPAS[®] is fitness-for-purpose. This means that the standard of accuracy required is based on an uncertainty that is independently determined to be appropriate for the analysis in question. A hypothetical laboratory performing exactly according to this predetermined standard will obtain z-scores like a random selection from a normal distribution. However, most laboratories will use methods with both a bias and a repeatability standard deviation that differs from the fitness-for-purpose uncertainty. Accordingly, the collected z-scores from a FAPAS[®] PT often deviate from the normal distribution. The deviation may take the form of heavy tails and outliers and, occasionally, asymmetry or multimodality. Because the scoring is based on an independently-prescribed uncertainty, it is logical to interpret z-scores on the basis of the normal distribution.

The properties of a normal distribution are such that about 95% of observations lie between ± 2 standard deviations. Performance in a FAPAS[®] PT, therefore, is considered fit-for-purpose if a z-score lies within the range ± 2 . It follows that an exactly-conforming participant's z-scores will fall outside

this range with a probability of 1 in 20. Occasional scores in the range $2 < |z| < 3$ may therefore be of no importance. Such z-scores require consideration and appropriate action. However, the probability of a conforming participant's z-score falling outside $|z| > 3$ is less than about 1 in 300. Such scores therefore represent results that are probably not fit-for-purpose and should be used to trigger investigation and remedial action.

Note. In the past, terms such as 'satisfactory', 'questionable' and 'unsatisfactory' have been applied to z-scores within certain ranges. This approach categorises the z-score when it is not appropriate to do so and is likely to be misleading. The limits $z = \pm 2$, $z = \pm 3$, must not be regarded as strict boundaries but should be treated as action limits. z-Scores are statistics and MUST be interpreted as such [15].

Note on homogeneity and z-scores. The requirement of distribution units to be 'sufficiently homogeneous' means that any variation detected between the units by the homogeneity test should be of negligible magnitude in relation to fitness-for-purpose and thus too small to affect z-scores. FAPAS[®] therefore takes no account of between-unit uncertainty in its scoring.

4.2.2. Interpreting Q-scores

This method of scoring has the disadvantage that the significance of any result is not immediately apparent. Q-scores cannot and *must not* be interpreted in the same way as z-scores.

Q-scores are not based on fitness-for-purpose. They are only an indication of the bias of each result relative to all of the submitted results.

FAPAS[®] will only ever issue such scores on rare occasions and then only as indicative measures and not to be used for performance evaluation purposes.

4.3. Appeals

FAPAS[®] undertakes to correct any mistakes attributable to errors on its part promptly and sympathetically. If a participant has any concerns about any aspect of the PT they should contact FAPAS[®] by email in the first instance. An investigation will be conducted in accordance with our management system and the participant advised of the outcome.

5. REFERENCES

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