ESTABLISHING A PROFICIENCY TESTING SCHEME TO ASSESS LABORATORY PERFORMANCE FOR PCDD/F AND PCB ANALYSIS OF FOODS.

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Introduction

Scientific judgements on food safety and quality are under continuous scrutiny by consumers and the media. There is therefore a continuing need for laboratories to demonstrate performance and reliability in laboratory analyses. Although there is much competition in the area of laboratory analysis, where customers demand lower prices, quality of laboratory procedures must not be compromised. It is now widely accepted in most areas of food analysis and regulation that there are three essential elements to laboratory quality assurance. These elements are:

- Use of validated analytical methods
- Accreditation to EN45000, involving third party auditing [1,2,3]
- Participation in laboratory proficiency testing (PT).

Almost all laboratories conducting PCDD/F and PCB analysis practise the first of these. The second is adopted by some and is becoming more common. The third of these, PT, is not widely used and up until now has not been available for laboratories conducting food analysis. Various inter-laboratory studies have been conducted such as 'Dioxins in Food' organised by the Norwegian Institute of Public Health[4] but this type of exercise does not use statistics suitable for the assessment of the performance of a laboratory. PT schemes provide an independent and unbiased assessment of performance, which can be used by participants to assess the performance of their analytical procedures.

Additional Measures Food Control Directive 93/99/EEC[5] describes quality standards for food control laboratories in the European Union and mandates participation in PT schemes. The endorsement of PT is such that principles by which such testing should be organised have been internationally agreed and are stipulated in an ISO/IUPAC/AOAC[6] International Harmonised Protocol.

This scheme will be organised by the Food Analysis Performance Assessment Scheme (FAPAS®) which was established in 1990 by the UK Ministry of Agriculture Fisheries and Food, specifically to organise PT for food analysis. It is the largest proficiency testing scheme of its kind with more than 800 participants from over 60 countries.

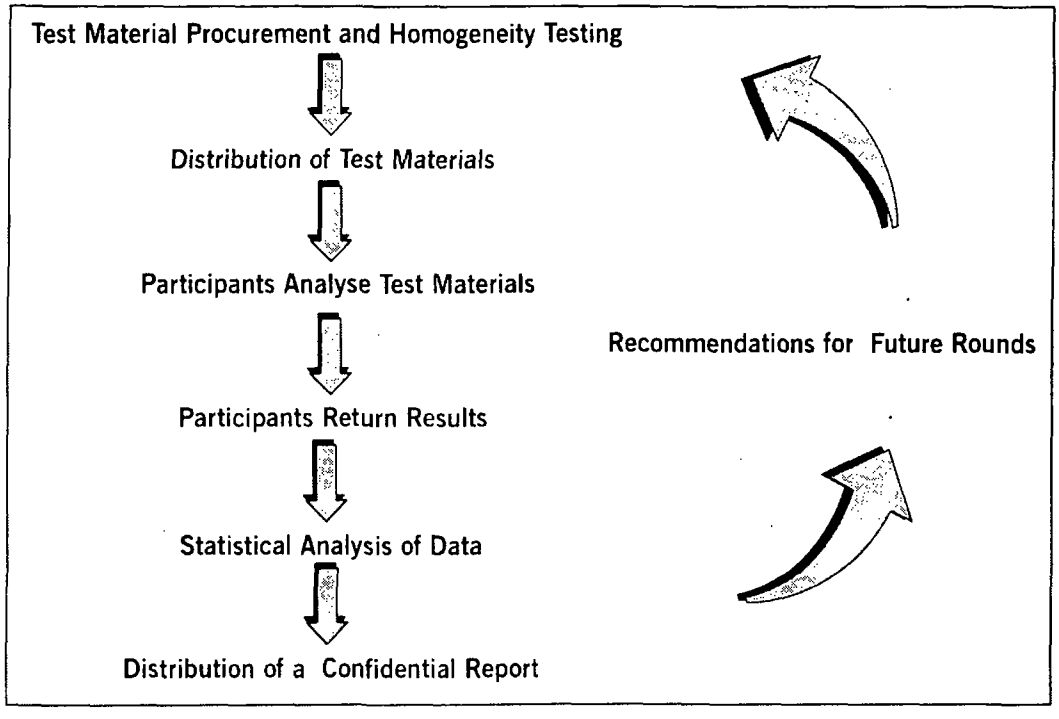
Establishing the scheme

Initially 1 or 2 test materials (depending upon response) will be distributed each year. The first of these will be a fish oil; the second will be a freeze dried fish or milk powder. These matrices were

chosen since they reflect sample types frequently analysed for these compounds. They will be realistic food-based test materials containing analytes at typical levels. PCDD/Fs and PCBs will not be added to the samples. The process is shown in Figure 1.

An International Advisory Committee consisting of members drawn from food industry, research organisations, regulatory bodies and food retailers will review and advise on the scientific aspects of this scheme, as required for any PT scheme.[6]

Figure 1: A Flow Diagram of the PT Process



Test Materials and Analytes

The suitability and quality of the test materials distributed are important to the effectiveness of a PT scheme. The two main criteria for a suitable test material are:

- It is important to choose test materials which resemble real samples as closely as possible. The ideal test materials are therefore real food samples naturally contaminated with the analyte in question.
- Successful PT and the comparison of participants' results can only take place if each participant receives an identical test material. The International Protocol[6] stipulates a strict homogeneity testing procedure by which randomly selected units from a fully homogenised bulk test material are tested for each relevant analyte. Only when the resulting data pass the appropriate statistical tests should the test material be used for a proficiency test. The F-test or the s_s/σ test, where s_s is the square route of the sampling variance and σ is the target value for

standard deviation[6] are used for this purpose. If neither of these tests give a satisfactory result, then the test material cannot be issued.

Performance Assessment

The assessment of a laboratory's performance is given by an assigned z-score which is defined as:

$$z = (x - \hat{X})/\sigma$$

Where x is the measurement of analyte concentration in the test material,

$\hat{X}$ is the assigned value, the best estimate of the "true" concentration of the analyte

and σ is the target value for standard deviation.

The assigned value, $\hat{X}$, is usually obtained from the robust mean[7] of the entire data population for the analyte in question. Target values for standard deviation (σ) are obtained from collaborative trial data (where available), from the use of the Horwitz equation[8] or from the use of the modified Horwitz equation if the concentration of the analyte concentration in question is <120 µg/kg.[9]

This PT scheme will assess (i) each PCDD/F and PCB congener assigned a WHO-TEF, (ii) each of the ICES 7 PCB environmental indicator congeners, (iii) the total WHO-TEQ, and the contribution to the WHO-TEQ from (iv) PCDD/Fs only, (v) non-ortho PCBs only (vi) ortho-PCBs only and (vii) the sum of the ICES 7 PCBs. This will mean that a total of 40 z-scores will be assigned for this exercise. Laboratories may participate in any or all of these evaluations.

Results

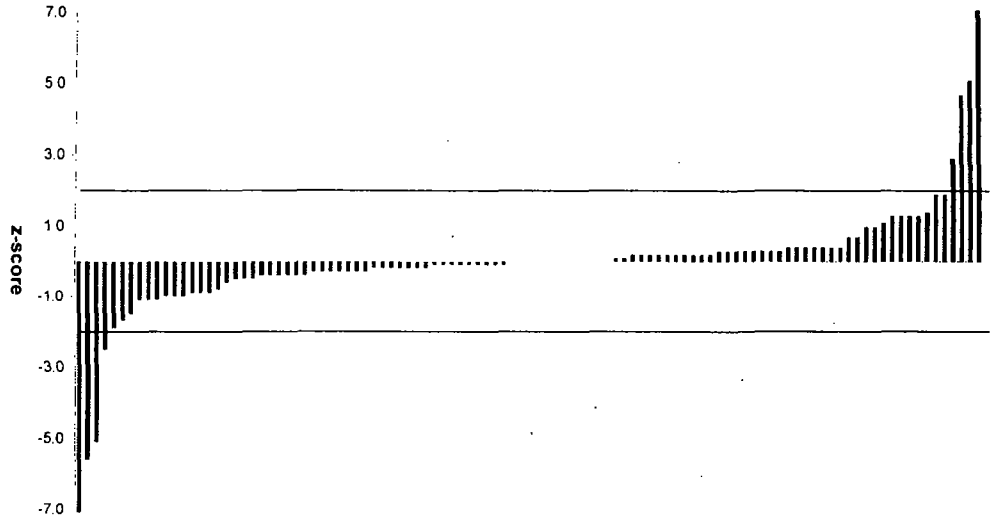
Reports for individual participants will be prepared and laboratories will be assigned a z-score for each result they submit (see above). z-Score markings will be shown in both tabular and ordered histogram form. A typical example of the histogram presentation for results submitted for a different test is shown in Figure 2. In this example, 97 laboratories were in the z-score range between +2 and -2 which is deemed as being satisfactory.[6,10]

In PT, each laboratory is free to choose a method of analysis. It is therefore possible to make correlations between methods used and performance. The identities of laboratories taking part in PT remain confidential at all times. Participation in a PT scheme is an indication of a commitment to quality. It is important therefore to understand the limitations of this external means of quality assessment when assessing the competence of a laboratory. If it is assumed that the assigned value ($\hat{X}$) and the target standard deviation (σ) correctly describe the variation of normally distributed participants' results (x), the data are known as "well behaved". In such cases there is a 1 in 20 chance that a participant's well behaved result may have a z-score of $|z|$ between 2 and 3, and about a 1 in 300 chance that it may have a z-score of $|z| > 3$.

Timetable and participation

The first round of testing will be for fish oil and is scheduled for February 2002. The performance of laboratories will be reported in April 2002. All laboratories worldwide are invited to participate. Larger numbers of participants will result in improved statistics. Details can be found by contacting the authors.

Figure 2: A Typical z-Score Histogram



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