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Analytical Quality Control Scheme
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Particle Size Analysis
Annual Report

2013/2014 (Year 20)

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PARTICLE SIZE ANALYSIS COMPONENT REPORT FROM THE CONTRACTOR

SCHEME OPERATION – YEAR 20 – 2013/14

1. Introduction

The National Marine Biological Analytical Quality Control (NMBAQC) Scheme addresses three main areas relating to benthic biological data collection:

- The processing of macrobenthic samples;
- The identification of macrofauna;
- The determination of physical parameters of sediments.

The twentieth year of the Scheme (2013/14) followed the format of the nineteenth year. A series of exercises involved the distribution of test materials to participating laboratories and the centralised examination of returned data and samples.

In the Year 20 NMBAQC Scheme fourteen out of fifteen registered laboratories participated in the particle size analysis exercises PS48, PS49, PS50 and PS51; four were government laboratories and ten were private consultancies. Six of the participants were responsible for CSEMP (Clean Seas Environment Monitoring Programme) sample analysis. To reduce potential errors and simplify administration, LabCodes were assigned in a single series for all laboratories participating in the benthic invertebrates, fish and particle size components of the NMBAQC Scheme (due to Thomson Unicomarine administering these three components).

As in previous years, some laboratories elected to be involved in limited aspects of the Scheme. CSEMP laboratories were required to participate in all relevant components of the Scheme, although this was not strictly enforced.

1.1 Summary of Performance

This report presents the findings of the Particle Size Analysis components for the twentieth year of operation of the National Marine Biological Analytical Quality Control (NMBAQC) Scheme. This component consisted of one module with four exercises:

- Analysis of four sediment samples (PS48, PS49, PS50 and PS51) for physical description.

The analytical procedures of this module were the same as for the nineteenth year of the Scheme. The results for the four exercises are presented and discussed. Comments are provided on the performance for each of the participating laboratories in each of the exercises.

In previous years the Particle Size exercises (PS) 'Pass/ fail' criteria were based upon z-scores from the major derived statistics with an acceptable range of ±2 standard deviations (see Description of the Scheme Standards for the Particle Size Analysis Component). The annual report for Scheme Year 16 deemed the use of z-scores inappropriate for such a low number of data returns where two erroneous results can significantly alter the 'Pass/ fail' criteria.

The z-score method also assumes that the majority of respondents are correct and raised genuine concerns regarding technique and method bias. Following this, the 'Pass/ fail' criteria are currently under review and alternative flagging criteria are being trialled. Therefore, Scheme Year 20 continues the use of z-scores calculated for each half-phi interval, and multivariate analysis using Euclidean distance matrices (dendrograms and non-metric MDS plots) from Year 19.

The variation within the ten replicate results produced for TUM in-house analysis (using the NMBAQC PSA SOP) was minimal for each of the four exercises; this is partly attributable to the use of only Malvern laser instruments and some standardised protocols, i.e. no use of chemical dispersants or hydrogen-peroxide pre-treatment. In most cases there was reasonably good agreement between participant laboratories for all four PS exercises. All four particle size exercises of Scheme year 20 received fourteen data returns; exercise 1 (PS48; Muddy Sand), exercise 2 (PS49; Gravelly Sand), exercise 3 (PS50; Sand) and exercise 4 (PS51; Gravel).

Comments are provided on the individual performance of the participating laboratories in each of the above components. A summary of their performance with respect to standards determined for the CSEMP is presented.

1.1.1.1 Statement of Performance

Each participating laboratory received a 'Statement of Performance', which included a summary of results for each of the Schemes modules and details the resulting flags where appropriate. These statements were first circulated with the 1998/1999 annual report, for the purpose of providing proof of Scheme participation and for ease of comparing year on year progress.

2. Summary of PSA Component

2.1 Introduction

The Particle Size Analysis (PS) module is described in more detail below. A brief outline of the information to be obtained from the module is given, together with a description of the

preparation of the necessary materials and brief details of the processing instructions given to each of the participating laboratories.

2.1.1 Logistics

The labelling and distribution procedures employed previously have been maintained and specific details can be found in the Scheme's annual reports for 1994/95 and 1995/96 (Unicomarine, 1995 & 1996).

2.1.2 Data returns

Return of data to Thomson Unicomarine Ltd. followed the same process as in previous years. Spreadsheet based forms (tailored to the receiving laboratory) were distributed for each circulation via email. All returned data have been converted to Excel 2003 format for storage and analysis. In this Scheme year data returns were prompt for most exercises, with reminders being distributed shortly before each exercise deadline.

2.1.3 Confidentiality

To preserve the confidentiality of participating laboratories, each are identified by a four-digit Laboratory Code. In September 2013 each participant was given a confidential, randomly assigned Scheme year nineteen LabCode. Codes are prefixed with the Scheme year to reduce the possibility of obsolete codes being used inadvertently by laboratories, e.g. Laboratory number four in Scheme year twenty will be recorded as LB2004.

In this report all references to Laboratory Codes are the post-August 2013 codes (Scheme year twenty). To reduce potential errors and simplify administration, LabCodes were assigned in a single series for all laboratories participating in the benthic invertebrate, fish and particle size components of the NMBAQC Scheme (due to Thomson Unicomarine administering these three components).

2.2 Particle Size Analysis (PS) Module

2.2.1 Description

This module examined the percentage of sediment found in each half-phi interval from the particle size analysis of replicate sediment samples. Four samples of sediment; two fine (PS48 and PS50) and two coarse (PS49 and PS51) were distributed in 2013/14. The sets of PS48 and PS50 replicate samples were derived from natural marine sediments; PS51 replicates were prepared from commercial aggregates; PS49 replicates were prepared from a combination of artificial and natural sediments; they were prepared as described below. In each case a random subsample of the prepared replicates was divided for laser diffraction analysis to ensure sample replicate consistency. To produce benchmark data, the replicates for PS48 and PS50 were analysed using a Malvern Mastersizer 2000; for PS51, the replicates were analysed using a Endecotts sieve shaker to produce benchmark data; and for

PS49, the replicates were analysed using a combination of sieve and laser analysis to produce benchmark data.

2.2.1.1 Preparation of the Sample

The sediment for PS48 was collected from the natural marine environment (a combination of Harwich estuarine sublittoral sediment and offshore in the southern North Sea). PS49 was created using a combination of commercially acquired aggregates, and sand from offshore in the southern North Sea. PS50 was created from a combination of sand from offshore in the southern North Sea and commercially derived sand. The final circulation (PS51) was created solely from commercially acquired materials.

All distributions containing naturally derived sand were screened at 1mm. Following sieving, the naturally derived sand portion was well mixed in a large tray and allowed to settle for a week. The sediment was sub-sampled by coring in pairs. One core of a pair was stored as the 'A' component, the other as the 'B'. To ensure sufficient weight for analysis, and to further reduce variation between distributed PS samples, this process was repeated three times for each sample replicate, i.e. each distributed sample was a composite of three cores.

The mud portion of the PS48 distribution was wet sieved at 63µm. An empty container was weighed before the wet sediment was decanted in to it. The sediment was left to settle for two days before the excess water was removed. The weight of the empty container was then subtracted from the full container and divided equally among the ten TUM samples and fifteen registered participants.

PS49 and PS50 replicates were produced by combining known quantities of commercially acquired aggregates and known quantities of naturally derived sediment.

PS51 replicates were produced by combining known quantities of commercially acquired aggregates. PS51 distributions were dry sieved and each phi interval separated by weight in to individual containers.

The numbering of the replicate samples was random. All of the odd-numbered 'B' components (a total of 10) were analysed in-house to assess the degree of inter-sample variation and produce benchmark data. All of these replicates were analysed using a Malvern Mastersizer 2000 laser. The 'A' components were assigned to participating laboratories randomly and distributed according to the Scheme timetable.

2.2.1.2 Analysis required

The participating laboratories were required to conduct particle size analysis on the samples following the NMBAQC's best practice guidance for particle size analysis to support biological data (Mason, 2011), either in-house or using a subcontractor. A written description of the sediment characteristics was to be recorded (pre-processing and post-processing using the Folk Triangle) as well as the %< 63µm and an indication of any peroxide treatment or chemical dispersant used. Also requested was a breakdown of the particle size distribution of the sediment, to be expressed as a weight or percentage of sediment in half-phi (ϕ) intervals. Optional data on the mean, median, sorting and skewness from the GRADISTAT program could also be provided. Approximately nine weeks were allowed for the analysis of each PS sample (PS48, PS49, PS50 and PS51).

2.2.2 Results

2.2.2.1 General comments

Fifteen laboratories subscribed to the PSA module for Scheme Year 20 (2013/2014). Fourteen of these participants submitted data returns.

Scheme Year 20 continues with the workbook method used in Year 19; this workbook auto-filled the "Final Merged Data" tab based on data which the laboratories provided for the sieve and laser analysis. The aim of this was to achieve more consistency in the way results were presented. Most participating laboratories now provide data in the requested format, though some variations remain. As previously reported, it should be remembered that the results presented may be from a more limited number of analytical laboratories than is immediately apparent since this component of the Scheme is often sub-contracted by participants to one of a limited number of specialist laboratories. For each of the four exercises fourteen of the fifteen participating laboratories returned data. Detailed results for each exercise have been reported to the participating laboratories (PS48, PS49, PS50 and PS51); additional comments are added below.

2.2.2.2 Analysis of sample replicates (benchmark data)

Replicate samples of the sediment used for the four PS distributions were analysed where required using a Malvern Mastersizer 2000 with Hydro-G Dispersion unit (no blue laser) to examine replicate variability and establish benchmark data. Replicates have been examined by both laser and sieve / pipette methods in earlier Scheme years; however as the majority of laboratories are conducting analyses by laser diffraction the testing of replicates is now undertaken using laser instruments only. Scheme Year 20 replicates follow the analysis method of Year 19 where all samples were analysed by Thomson Unicomarine Ltd.

Sample PS48 comprised of Muddy Sand sediment (average of 3.55% <63µm, mean phi of 1.58), the Malvern Mastersizer 2000 showed generally good agreement between replicate samples. However, some variability was present within the ten replicates (i.e. TUM02 - TUM05 showed percentage mud of 2% or below whereas the remaining replicates reported between 3% and 7%). The replicates were laser analysed only. Results for the individual replicates are provided in Table 1 and are displayed in Figure 1 (PS48 Report).

Sample PS49 comprised a Gravelly Sand sediment (average of 0.00% <63µm, mean phi of 0.29). The replicates used both sieve and laser analysis. The Malvern Mastersizer 2000 showed no real discernable variation between replicate samples. Results for the individual replicates are provided in Table 1 and are displayed in Figure 1 (PS49 Report).

Sample PS50 comprised a combination of naturally derived sand and commercially acquired sand (average of 2.5% <63µm, mean phi of 1.52). The replicates were analysed using laser only. The replicates show good agreement between each other. Results for the individual replicates are provided in Table 1 and are displayed in Figure 1 (PS50 Report).

Sample PS51 was comprised of Gravel (average of 0.00% <63µm, mean phi of -2.81). Replicates showed very good agreement between each other. The replicates were sieved only. Results for the individual replicates are provided in Table 1 and are displayed in Figure 1 (PS51 Report).

2.2.2.3 Results from participating laboratories

Where they were provided, summary statistics for the four PS circulations are presented in Table 1 in each individual exercise report (see PS48 Report, PS49 Report, PS50 Report and PS51 Report). After resolution of the differences in data format, the size distribution curves for each of the sediment samples were plotted and are presented in Figures 2 in each individual exercise report. Included in each of these figures, for comparison, are the mean distribution curves for the replicate samples as obtained by Thomson Unicomarine Ltd (PS48, PS49, PS50 and PS51), using Malvern Mastersizer 2000, where required.

Table 2 in each exercise report (see PS48 Report, PS49 Report, PS50 Report, PS51 Report) shows a summary of the z-scores calculated for each half phi interval. Intervals left blank or marked “not analysed” were entered as zero to calculate the z-scores. Figures 4 and 5 in PS48, PS49, PS50 and PS51 show the results of the cluster analysis. For each exercise ten out of the fourteen participating laboratories stated that they were following NMBAQC methodology, two laboratories were using a combination of other and NMBAQC methodologies and two laboratories stated that they were using other methods.

2.2.2.4 PS48 particle size distribution

There was generally good agreement for PS48 between the results from the analysis of replicates and those from the majority of participating laboratories (see Figure 2). One laboratory (LB2029) continued recording greater percentages of particles between phi intervals 2 and 7.5 compared to other laboratories. LB2007 did not record phi intervals between 4.5 and 8. There were also differences reported between 9 and 13.5 shown by this laboratory being the last to reach 100% on the cumulative curve. All but one (LB2007) of the participants used the laser diffraction technique to analyse the sample. Table 1 shows the variation in data received from the participating laboratories. The derived statistic for %mud for laboratories following the NMBAQC Scheme best practice guidance ranged from 8.71% to 45.31% (incl. results from LB2007 and LB2060), excluding data from the replicate analyses produced by Thomson Unicomarine Ltd (Malvern Mastersizer 2000). Those following other methodology ranged from 12.51% to 23.05%.

2.2.2.5 PS49 particle size distribution

There was generally good agreement for PS49 between the results from the analysis of replicates and those from the participating laboratories (see Figure 2). One laboratory (LB2056) recorded significantly higher proportions between phi intervals -2.5 and -2. Table 1 shows the variation in data received from the participating laboratories. The derived statistic for laboratories following the NMBAQC method for %mud ranged from 0.00% to 1.02% (incl. results from LB2007 and LB2060), excluding data from the replicate analyses produced by Thomson Unicomarine Ltd (Malvern Mastersizer 2000). One laboratory (LB2020) recorded 0.33% mud following other methodology.

2.2.2.6 PS50 particle size distribution

There was generally good agreement between the results from the analysis of PS50 replicates and those from the participating laboratories (see Figure 2). Two laboratories show displaced distribution curves (LB2029 and LB2031). LB2029 recorded a higher cumulative proportion of particles between phi intervals 0 and 3. LB2031 did not start recording results until phi interval 1.5 with a subsequent sharp rise in the percentage of particles between 1.5 and 3.5. Both laboratories were the only participants that recorded 100% sand. All but one (LB2007) of the participants analysed using laser diffraction techniques regardless of methodology used. Table 1 shows the variation in data received from the participating laboratories where data was submitted. The derived statistic for %mud ranged from 1.28% to 7.89% for participants following NMBAQC Scheme methodology (incl. results from LB2007 and LB2060), excluding data from the replicate analyses produced by Thomson Unicomarine Ltd (Malvern Mastersizer 2000) and from 1.42% to 3.18% for those following different methodology.

2.2.2.7 PS51 particle size distribution

There was generally good agreement between the results from the analysis of PS51 replicates and those from the participating laboratories (see Figure 2). Two out of the fourteen participating laboratories (LB2007 using NMBAQC methodology and LB2056 using other methods) analysed sediment below 1mm. LB2056 recorded intermittent sharp rises in the cumulative percentage curve. This was due to this laboratory recording at each whole phi rather than at each half phi. LB2020 did not start recording phi values until -3 leading to a sharp rise between phi values -3 and -2.5. Table 1 shows the variation in data received from the participating laboratories where data was submitted. Due to this exercise being sieved only, the derived statistic for laboratories following both the NMBAQC method and/or other methods for %mud was 0.00% for all participants, excluding data from the replicate analyses produced by Thomson Unicomarine Ltd (Malvern Mastersizer 2000).

2.2.3 Discussion

The samples distributed as PS48, from an analysis of replicates (Figure 1), appear to be good with very little variance. Results from participating laboratories (Figure 2) also showed a generally good consistency in similarity (omitting LB2029). Cluster analysis using Euclidean distance showed that six laboratories (LB2029, LB2007, LB2031, LB2057, LB2021 and LB2020) clustered away from the main group. The discrepancy in LB2029's data was characterised by the recording of greater values for percentage particles between phi intervals 2 and 7.5 than all other laboratories.

LB2007 did not report any data values between phi intervals 4.5 and 8. This laboratory also showed a greater degree of variability in recorded sediment proportions between phi intervals 9 and 13.5 compared to other laboratories. This laboratory also recorded the lowest percentage of mud (0.22%) compared to other laboratories. These results could be due to the methodology used by this laboratory to analyse the sediment.

The samples distributed as PS49, from an analysis of replicates (Figure 1), appear to be good with very little variance. Results from participating laboratories were also generally consistent with one another (Figure 2). Cluster analysis shows that five laboratories (LB2056, LB2022, LB2031, LB2029, and LB2003 clustered away from the other laboratories. LB2056's cumulative percentage curve (Figure 2) shows a sharp rise in percentage particles between phi intervals -2.5 and -2. LB2022 recorded a greater percentage of particles between -4 and -2.5. This laboratory also did not record values between phi intervals -2.5 and 0.00.

The samples distributed as PS50, from an analysis of replicates (Figure 1), appear to be good with very little variance. Results from participating laboratories were also generally consistent with one another (Figure 2). Cluster analysis using euclidean distance shows that four laboratories (LB2031, LB2029, LB2007 and LB2020) clustered away from the main

group. LB2031 did not start recording results until phi interval 1.5. This is shown on the cumulative percentage curve by a displacement of one phi. This laboratory also recorded higher percentage proportions between phi intervals 2.5 and 4. This result is likely attributed to LB2031 using sieve only analysis for particles below 1mm.

The samples distributed as PS51, from an analysis of replicates (Figure 1), appear to be good with very little variance. Results from participating laboratories were reasonably consistent with one another (Figure 2). LB2056 shows intermittent sharp rises in percentage proportions at each phi interval. This is due to the equipment this laboratory uses to analyse sediment. LB2020 recorded the majority of its sediment proportion at phi interval 2.5. Phi interval 2 is also abnormally low compared to all other laboratories. The reason for this apparent discrepancy is that this laboratory measures it's >1mm fractions according British Standard sieve mesh sizes rather than ISO standard. These mesh sizes are incompatible with one another in terms of assessing percentage proportions at half phi intervals and therefore the results shown do not accurately reflect the NMBAQC SOP.

Participating laboratories were asked to provide the sediment description using the Folk triangle post analysis. Data was provided by all fourteen participating laboratories for PS48, PS49, PS50 and PS51. Two laboratories (LB2020 and LB2022) failed to provide the post analysis description for PS48. For PS48, eight laboratories (LB2007, LB2015, LB2021, LB2027, LB2029, LB2054, LB2056 and LB2057) had post-analysis sediment descriptions of Muddy Sand; four laboratories (LB2003 , LB2031, LB2032 and LB2060) had a post-analysis description of Sand. Two laboratories (LB2020 and LB2022) failed to provide the post analysis description for PS49. For PS49, eleven participating laboratories recorded the post-analysis sediment description as Gravelly Sand. The remaining laboratory (LB2007) recorded Sandy Gravel. Four laboratories (LB2015, LB2020, LB2022 and LB2029) failed to provide the post analysis description for PS50. All other post-analysis sediment descriptions for PS50 were Sand. Four laboratories (LB2015, LB2020, LB2022 and LB2029) failed to provide the post analysis description for PS51. All other post-analysis sediment descriptions for PS50 were Gravel.

It is essential that analytical methods, including pre-treatment, are stated when reporting or attempting to compare results. The situation is further complicated by the fact that the difference between the techniques and the effects of the pre-treatment also varies with the nature of the sediment sample. As demonstrated in these and previous PS exercises, possible variations in equipment and methods can result in variable data. In order to eliminate as much variation as possible the NMBAQC's Best Practice Guide was devised for use in Scheme Year 17. Although most laboratories used the methods detailed in this document, a

few laboratories still used in-house methodologies. All laboratories involved in CSEMP sample analysis used the NMBAQC PSA SOP for supporting biological data.

The workbook format introduced in Scheme Year 19 is continued in Scheme Year 20, the aim of this is to standardise the way in which laboratories provide data. Over the four exercises most laboratories completed the forms correctly.

One of the issues that came to light during the analysis stage was that the half-phi percentage proportions in the 'PS_Final Merged Data' tab did not match the data from participant laser and sieve entries. This was due to an auto-calculation error in the distributed workbook. Therefore, participant results were calculated independently by using the provided sieve and laser data, where appropriate, supplied and re-calculated using the in-house PSA analysis excel workbook. The results contained in each report are based upon the independently merged data presented in Appendix 2 rather than those presented by the results from the 'PS_Final Merged Data tab' of each participant.

It is important for laboratories using the NMBAQC Scheme SOP for particle size analysis to adhere to it. There have been instances where some laboratories have modified how they analysed the sediment due to the apparatus (i.e. laser analysis equipment) used. Altering the SOP on the basis of laser equipment capabilities can change the final results derived from analysis.

The main issue with the workbook trialled in Scheme Year 18 was with the laser replicates section, where sediment ≥1mm was being passed through laser diffraction. If following the NMBAQC methodology, laser subsamples should be passed through a 1mm sieve before laser diffraction. Although this has not occurred in Scheme Year 20, it remains noteworthy if standardisation of submitted results is to be maintained.

PS51 was distributed as a sieve only exercise and therefore analysis below 1mm was not a requirement. However, some laboratories recorded results below 1mm. These results had to be discounted. Although there were trace amounts of sediment below 1mm, if following NMBAQC SOP, there was insufficient material (< 5%) to analyse the below 1mm portion. It is acknowledged, however, that those participants using other methods may not know of the NMBAQC SOP. This serves an example showing the need for participants to follow the same protocols during analysis to make the results more representative .

2.2.4 Application of NMBAQC Scheme Standards

One of the key roles of the Particle Size Analysis component of the NMBAQC Scheme is to assess the reliability of data collected as part of the Clean Seas Environment Monitoring Programme (CSEMP; formerly UK NMMP). With this aim performance target standards were defined for certain Scheme modules and applied in Scheme year three (1996/97). These standards were the subject of a review in 2001 (Unicomarine, 2001) and were altered in Scheme year eight; each performance standard is described in detail in the Description of the Scheme Standards for the Particle Size Analysis Component document. In previous years laboratories meeting or exceeding the required standard for a given exercise would be considered to have performed satisfactorily for that particular exercise. A flag indicating a 'Pass' or 'Fail' would be assigned to each laboratory for each of the exercises concerned. As the Pass/fail criteria are under review for the PS exercises in Scheme Year 20, a 'Pass' or 'Fail' flag will not be assigned to each lab for these particular exercises.

2.2.4.1 Laboratory Performance

Z-scores and cluster dendrogram figures are presented in each of the PS exercise reports; however these are only for illustration purposes. The investigations into new pass/fail standards are still underway. Pass/fail criteria will be introduced when sufficient data are collected using the new analysis guidance method.

3. Conclusions and Recommendations

A number of observations may be made from the results of the exercises described above. The following is a summary of the major points of importance.

1. Laboratories should endeavour to report their PS results in the requested format, *e.g.* at half phi intervals. This would enable the direct comparison of data from all participants and simplify the creation of cumulative curve figures. Participants should review their data prior to submission; zeros should only appear in submitted data where no material was present; dashes, '-', should appear where analysis has not been conducted.

2. Laboratories involved in CSEMP data submission should endeavour to return data on **ALL** necessary components of the Scheme in the format requested. This will be required to allow the setting of performance "flags". Non-return of data will result in assignment of a "Fail" flag. For CSEMP laboratories this deemed "Fail" for no submitted data is to be perceived as far worse than a participatory "Fail" flag.

3. Particle size exercises (PS) over the years have shown differences in the results obtained by different techniques (laser and sieve / pipette), in-house methods (*e.g.* pre-treatment) and also differences between equipment (*e.g.* Malvern Mastersizer 2000, Mastersizer X

and Coulter LS230 lasers). PS data indicates that the variance between laser and sieve results is further emphasised by certain sediments characteristics. The overall range of these variances needs to be determined if combining data sets derived from differing methods. The NMBAQC's Best Practice Guide has been developed for use in Scheme Year 17; this has helped to reduce the amount of variation between methods. It is essential that particle size data are presented with a clear description of the method of analysis and equipment used.

4. An improved learning structure to the Scheme through detailed individual exercise reports has been successfully implemented and was continued in this Scheme year. For the PS exercises, detailed results have been forwarded to each participating laboratory as soon as possible after the exercise deadlines. Participants that submit significantly incorrect data are contacted immediately to ensure that in-house checks can be implemented to ensure future quality assurance. The PS48, PS49, PS50 and PS51 reports included the data submission sheets received from all participants as an appendix; Participants are encouraged to review their exercise reports and provide feedback concerning content and format wherever appropriate.

5. The current NMBAQC Scheme standards for PSA are under review. The alternative use of z-scores for each phi-interval, trialled in Scheme Year 17 appears inappropriate for such a low number of data returns where two erroneous results can significantly alter the pass/fail criteria. For example, this can occur if laboratories do not have the representative sieves to analyse the whole range of sediment fractions. The z-score method also assumes that the majority of respondents are correct and raised genuine concerns regarding technique and method bias. Scheme Year 20 (2013/14) follows Year 19 in that z-score analysis was run alongside cluster analysis using Euclidean distance matrices.

6. Future reports could include a reverse ring test whereby benchmark samples are tested by a randomly selected laboratory to ensure representative results analysed for the ring test are satisfactory. This could also be achieved by the responsible laboratory providing raw data (.mea) to external verifiers to assess in-house quality. It is also possible to add SIMPROF testing of own samples to show minimal inter-sample variation in interim reports.

4. References

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