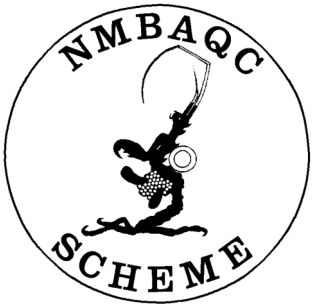

BEQUALM

NATIONAL MARINE BIOLOGICAL

ANALYTICAL QUALITY CONTROL SCHEME

Particle Size Component Report

Scheme Operation - Year 18 - 2011/2012

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PARTICLE SIZE ANALYSIS COMPONENT REPORT FROM THE CONTRACTOR

SCHEME OPERATION – YEAR 18 – 2011/12

Scheme Year 18 Exercise Reports (hyperlinked in this report)

Particle Size Results – PS40

Particle Size Results – PS41

Particle Size Results – PS42

Particle Size Results – PS43

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 eighteenth year of the Scheme (2011/12) followed the format of the seventeenth year. A series of exercises involved the distribution of test materials to participating laboratories and the centralised examination of returned data and samples. 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).

In the Year 18 NMBAQC Scheme eleven laboratories participated in the particle size analysis exercises PS40, PS41, PS42 and PS43; five were government laboratories; six were private consultancies. Five 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 eighteenth 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 for physical description (Particle Size module).

The analytical procedures of this module were the same as for the seventeenth 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. Scheme Year 17 trialled the use of z-scores calculated for each half-phi interval, Scheme Year 18 trials the use of multivariate analysis using Euclidean distance matrices (dendrograms and non-metric MDS plots).

The variation within the ten *replicate* results produced by the benchmark laboratories using the NMBAQC PSA SOP was minimal for PS40-43; 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. The first particle size exercise of the Scheme year (PS40; sandy mud sample) received eleven data returns. The second particle size exercise of the Scheme year (PS41; sand sample) received eleven data returns. The third particle size of the Scheme year (PS42; artificial gravel sample) received eleven data returns. The final particle size exercise of the Scheme year (PS43; gravelly muddy sand sample) received eleven data returns.

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

There is one module in the particle size component; Particle Size Analysis (PS) module.

This 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). Email was the primary means of communication for all participating laboratories. This has considerably reduced the amount of paper required for the administration of the Scheme.

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, with additional hard copies where appropriate. All returned data have been converted to Excel 2003 format for storage and analysis. In this and previous Scheme years slow or missing returns for exercises lead to delays in processing the data and resulted in difficulties with reporting and rapid feedback of results to laboratories. Reminders were 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 2010 each participant was given a confidential, randomly assigned Scheme year eighteen 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 eighteen will be recorded as LB1804.

In this report all references to Laboratory Codes are the post-August 2010 codes (Scheme year eighteen). 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 component 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 (PS40 and PS41), one coarser (PS42) and one mixed (PS43) were distributed in 2011/12. The sets of PS40 and PS41 replicate samples were derived from natural marine sediments; PS42 replicates were artificially prepared from commercial aggregate materials; PS43 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 were divided for laser diffraction analysis to ensure sample replicate consistency. For PS40 - 43 the *replicates* were analysed where required using a Malvern Mastersizer 2000 to produce benchmark data.

2.2.1.1 Preparation of the Samples

The first two PS circulations were sediments collected from natural marine environments (Harwich for PS40, Milford Haven for PS41); the third (PS42) was artificially created from commercially acquired materials; the final sediment (PS43) was artificially created from natural sediments from Harwich and commercially acquired materials. Natural material for PS40 and PS41 was returned to the laboratory and coarse sieved (1 mm) to remove gravel, shell and large faunal content. A minimum of 30 litres of visually similar sediment was collected. Following sieving, the sediment for PS circulation 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 artificial PS42 replicates were produced by combining known quantities of commercially acquired material. For the PS43 replicates, known quantities of commercially acquired gravel were added to known quantities of mud from PS40 (Harwich).

The numbering of the replicate samples was random. All of the odd-numbered 'B' components (a total of 14) were sent for particle size analysis to assess the degree of inter-sample variation and produce benchmark data. All of these *replicate*s 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 ( ) 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 (PS40, PS41, PS42 and PS43).

2.2.2 Results

2.2.2.1 General comments

Eleven laboratories subscribed to the exercises in 2011/12.

For Scheme year 18 a new workbook was provided for laboratories; this auto-filled the "Final Merged Data" tab based on what laboratories provided for the sieve and laser data. 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 all of the eleven participating laboratories returned data; Detailed results for each exercise have been reported to the participating laboratories (PS40, PS41, PS42 and PS43); 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. In Year 16, half the *replicates* were analysed using a Malvern Mastersizer 2000 laser and half by a Malvern Mastersizer X log bed laser. In Year 17 *replicate* analyses were performed by Plymouth University, Geography Department (Malvern Mastersizer 2000) (PS40 and PS41) and Thomson Unicomarine Ltd (Malvern Mastersizer

2000) (PS42 and PS43). *Replicate* samples analysed by both laboratories showed very good agreement. In Year 18 all replicates were analysed by Thomson Unicomarine Ltd.

Sample PS40 comprised of sandy mud sediment (average of 86.40% <63*μ*m, mean phi of 6.46), the Malvern Mastersizer 2000 showed good agreement between *replicate* samples. One replicate (PS40_1869) had a slightly higher percentage of silt and lower percentage of sand compared to the other replicates causing the post-analysis description of this replicate to be Mud, rather than Sandy Mud. Results for the individual *replicates* are provided in Table 1 and are displayed in Figure 1 (PS40 Report).

Sample PS41 comprised a sand sediment (average of 9.41% <63*μ*m, mean phi of 2.58). The Malvern Mastersizer 2000 showed no real discernable variation between *replicate* samples. Two of the replicates (PS41_60 and PS41_62) had slightly higher percentages of silt and lower percentages of sand causing them to be classified as Muddy Sand rather than Sand in the post analysis description. Results for the individual *replicates* are provided in Table 1 and are displayed in Figure 1 (PS41 Report).

Sample PS42 comprised of an artificial gravel sediment (average of 0.00% <63*μ*m, mean phi of -2.93). The replicates were analysed by dry sieving only, no laser analysis was required. The replicates show good agreement between each other. Replicate PS42_64 has a slightly higher percentage at -3.5 phi compared to the other replicates. Results for the individual *replicates* are provided in Table 1 and are displayed in Figure 1 (PS42 Report).

Sample PS43 comprised of Muddy Sandy Gravel sediment (average of 25.59% <63*μ*m, mean phi of 0.646). Replicates showed fairly good agreement, there was a range of approximately 10% in the amount of sediment <0 phi. Results for the individual replicates are provided in Table 1 and are displayed in Figure 1 (PS43 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 PS40 Report, PS41 Report, PS42 Report, PS43 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 (see PS40 Report, PS41 Report, PS42 Report, PS43 Report). Included in each of these figures, for comparison, are the mean distribution curves for the *replicate* samples as obtained by Thomson Unicomarine Ltd (PS40, PS41, PS42 and PS43), (using Malvern Mastersizer 2000, where required). Table 2 in each report (see PS40 Report, PS41 Report, PS42 Report, PS43 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. Each individual exercise report also provides a bar-chart of each lab's z-scores. Figures 7 – 10 in PS40 and PS41 and Figures 5 and 6 in PS42 and PS43 show the results of the cluster analysis. For each exercise nine out of the eleven participating laboratories stated that they were following the NMBAQC's methods. Two laboratories (LB1814 and LB1830) stated that they were using alternate methods.

2.2.2.4 Fortieth distribution – PS40

There was generally good agreement for PS40 between the results from the analysis of *replicates* and those from the majority of participating laboratories (see Figure 2). One lab (LB1830) had missing data values for some of the half-phi intervals towards the end of the data set. All 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 %silt for those laboratories following the NMBAQC methods ranged from 78.99% to 92.00%, excluding data from the *replicate* analyses produced by Thomson Unicomarine Ltd (Malvern Mastersizer 2000). The two laboratories (LB1814 and LB1830) following alternate methods recorded a %silt of 90.38% and 86.32% respectively.

2.2.2.5 Forty-first distribution – PS41

There was generally good agreement for PS41 between the results from the analysis of *replicates* and those from the participating laboratories (see Figure 2). Of the laboratories following the NMBAQC methods six (LB1802, LB1803, LB1806, LB1809, LB1811, and LB1818) stated that they used laser diffraction only to analyse the sample; LB1801 and LB1804 used sieves and laser diffraction. One laboratory (LB1816) only provided data in the "Final Merged Data" tab of the worksheet, as there is data greater than 1mm it will be assumed that a combination of sieving and laser diffraction were used to analyse the sample. Of the laboratories not following NMBAQC methods LB1830 only used laser diffraction and LB1814 used sieves and laser diffraction. Table 1 shows the variation in data received from the participating laboratories. The derived statistic for laboratories following the NMBAQC methods for %silt ranged from 6.39% to 18.94%, excluding data from the *replicate* analyses produced by Thomson Unicomarine Ltd (Malvern Mastersizer 2000). The two laboratories (LB1814 and LB1830) following alternate methods recorded a %silt of 12.27% and 15.07% respectively.

2.2.2.6 Forty-second – PS42

There was generally good agreement for PS42 between the results from the analysis of *replicates* and those from the participating laboratories (see Figure 2). Seven out of the nine laboratories following the NMBAQC methodology (LB1801, LB1802, LB1803, LB1806, LB1809, LB1816 and LB1818) used dry sieving only to analyse the sample. The remaining

two laboratories (LB1804 and LB1811) attempted laser diffraction as well as dry sieving but found there was insufficient sediment to do more than one run through the laser. Two participating laboratories used alternate methods; LB1814 used dry sieves from -6.5 to 4.0 phi and LB1830 dry sieves from -6.5 to 0 phi. Two laboratories (LB1816 and LB1830) did not provide the data in half phi intervals. Table 1 shows the variation in data received from the participating laboratories where data was submitted. The derived statistic for the % silt was 0% for all laboratories except for those (LB1804 and LB1811) who attempted laser diffraction. The %silt for these two laboratories was 0.07% (LB1804) and 0.13% (LB1811).

2.2.2.7 Forty-third distribution – PS43

There was a fair amount of variation between the results from analysis of replicates and those from the participating laboratories (see Figure 2). Ten laboratories (LB1801, LB1802, LB1803, LB1804, LB1806, LB1809, LB1811, LB1814, LB1816 and LB1830) used sieve and laser analysis to analyse the sample; one lab (LB1818) only used laser analysis. LB1806 only recorded above -2.5 , displacing their cumulative curve by 2 phi at the beginning. The stone that the majority of laboratories recorded at -4.5 to -4.0 phi was recorded half a phi out by LB1801 and one phi out by LB1802. Table 1 shows the variation in data received from the participating laboratories where data was submitted. For participating laboratories using the NMBAQC method the derived statistic for the % silt ranged from 1.39% to 89.396%, excluding data from the *replicate* analyses produced by Thomson Unicomarine (Malvern Mastersizer 2000). The two laboratories (LB1814 and LB1830) following alternate methods recorded a %silt of 24.80% and 34.50% respectively.

2.2.3 Discussion

The samples distributed as PS40 appeared from an analysis of *replicates* (Figure 1) to be good replicates with very little variance. Results from participating laboratories (Figure 2) showed a general similarity in distribution curves. Cluster analysis using Euclidean distance showed that three laboratories (LB1802, LB1806 and LB1830) clustered away from the majority of laboratories. Of these three laboratories, one (LB1830) was using an alternate method rather than the NMBAQC methodology. The main discrepancy in LB1830's data was that there were missing data entries at 8.0 to 8.5 phi and 9.0 to 9.5 phi. The data did not total 100% in the final merged data sheet as the laser data included values for sediment >1mm which should have been removed and the laser data re-scaled. LB1806 used a hydrogen peroxide pre-treatment; this did not appear to have any effect their data in comparison to those laboratories who did not use a pre-treatment. LB1806 did not record any particles smaller than 9.0 phi (1.381μm). LB1802 did not record any particles smaller than 8.0 phi (3.906μm) and recorded the highest percentage sand (21.01%). LB1802 did not rescale laser reps 2 and 3, however the percentage of >1mm detected by the laser was minimal.

The samples distributed as PS41 appeared from an analysis of *replicates* (Figure 1) to be good replicates with little variance. Results from participating laboratories were generally concurrent (Figure 2), although six participating laboratories (LB1801, LB1806, LB1809, LB1811, LB1818 and LB1830) did not remove laser data greater than 1mm, consequently the laser data was not re-scaled. Due to the high number of errors occurred in the workbooks analysis becomes problematical. The MDS plots (Figure 8) from the cluster analysis show that the participating laboratories are fairly spread out indicating that the laboratories are not closely related. It is important to note that the box surrounding the MDS plots is not an axis and does not represent any form of scale. The dendrogram in Figure 7 shows six SIMPROF cluster groups; three of which only include single laboratories (LB1814, LB1816 and LB1818), two cluster groups include two laboratories (LB1802 and LB1806 form one, LB1803 and the TUM AVERAGE form the other) and one group contains five laboratories (LB1809, LB1830, LB1804, LB1801 and LB1811).

The samples distributed as PS42 appeared from an analysis of *replicates* (Figure 1) to be good replicates with little variance. Results from participating laboratories were generally in accord (Figure 2). Cluster analysis based on a Euclidean distance matrix produced six cluster groups. Cluster group A comprised of one laboratory (LB1806). The cumulative percentage curve in Figure 2 shows that the LB1806 curve differs greatly from the other participating laboratories as they did not measure any sediment greater than -2.5 phi (4.0mm). Cluster group B is formed of two samples (LB1816 and LB1830); both these laboratories did not provide data in half phi intervals. LB1814 and LB1811 grouped into separate cluster groups C and D, respectively. LB1814 used an alternate method to the NMBAQC methodology, and recorded a much higher percentage at -4.5 phi compared to other participating laboratories. LB1811 recorded a lower percentage of sediment at -3.0 phi compared to other laboratories. Cluster group E was the largest group consisting of four participating laboratories (LB1801, LB1803, LB1804 and LB1809) and the benchmark data (TUM AVERAGE). Cluster group F consisted of two laboratories (LB1802 and LB1818) and was closely related to cluster group E. All these seven laboratories follow very similar cumulative percentage curves (Figure 2).

The samples distributed as PS43 appeared from an analysis of *replicates* (Figure 1) to be good replicates with little variance. The main source of variation was found in the percentage of sediment less than 1mm (0 phi), this varied from 20.97% to 28.82%. Cluster analysis from a Euclidean distance matrix produces six SIMPROF cluster groups with four groups consisting of a single laboratory. Cluster group A is formed of a single lab (LB1802).The cumulative percentage curves in Figure 2 show that the stone that most laboratories recorded at -4.5phi has been recorded one phi out at -3.5phi by LB1802. LB1802 also recorded the highest percentage of greater than 1mm. Cluster group B comprised of two laboratories, LB1816 and LB1818. LB1818 only ran laser analysis on the sample and so did not record any particles greater than 1mm. They later contacted Thomson Unicomarine to explain that they had

forgotten to include the sieve data. LB1816 stated they followed the NMBAQC PSA SOP but also declared that the sample was wet sieved through a 2mm sieve rather than a 1mm. This would explain the plateau in their cumulative percentage curve (Figure 2) between -1 and 0 phi (2000-1000μm). LB1816 also recorded the second lowest percentage of greater than 1mm sediment. Cluster group C comprised of one lab, LB1806. The cumulative percentage curve in Figure 2 shows that LB1806 produced the second highest percentage of sediment greater than 1mm. LB1806 did not analyse any sediment larger than -2.5phi (5600μm), therefore the stone at -4.5phi has been displaced to -2.5phi. Cluster group D comprised of LB1801. This lab had the third highest percentage of sediment greater than 1mm and the stone that the majority of laboratories recorded at -4.5phi, they recorded at -5.0phi. Cluster group E was formed of one lab, LB1830. This lab was relatively similar to cluster group F but recorded the greater than 1mm at one phi intervals rather than half-phi intervals. Cluster group F comprised of five participating laboratories (LB1804, LB1809, LB1803, LB1811 and LB1814) and the Benchmark Data (TUM AVERAGE). All these laboratories recorded the stone at -4.5phi. Four of the laboratories (LB1803, LB1804, LB1809 and LB1811) had very similar percentages of sediment greater than 1mm, ranging from 58.54% to 61.86%. LB1814 and the Benchmark Data had slightly higher percentages the sediment greater than 1mm at 71.09 and 70.30, respectively.

Participating laboratories were asked to provide the sediment description using the Folk triangle post analysis. Data were provided by all eleven participating laboratories for PS40, PS41 and PS42. Two laboratories (LB1801 and LB1806) failed to provide the post analysis description for PS43. For PS40, four laboratories (LB1802, LB1809, LB1811 and LB1830) had a post-analysis description of Sandy Mud. Other post-analysis descriptions included Silt (LB1801), Slightly Sandy Mud (LB1803), Mud (LB1804 and LB1814), Sandy Silt (LB1806), Medium Silt (LB1816) and Muddy Sand (LB1818). For PS41, five participating laboratories (LB1804, LB1809, LB1811, LB1814 and LB1818) recorded the post-analysis sediment description as Muddy Sand, three laboratories (LB1802, LB1803 and LB1806) defined the sediment as Sand, two laboratories (LB1801 and LB1816) defined the sample as Fine Sand and LB1830 recorded the sediment description as Silty Sands. All post-analysis sediment descriptions for PS42 were Gravel except for LB1816 who recorded a sediment type of Moderately Well Sorted Fine Gravel. For PS43 two laboratories (LB1801 and LB1806) did not provide post-analysis sediment descriptions. Three laboratories (LB1803, LB1804 and LB1809) described the sediment as Gravelly Muddy Sand; two laboratories (LB1811 and LB1814) described the sediment as Muddy Sandy Gravel. Other post-analysis sediment description included Sandy gravel (LB1802), Very poorly sorted very fine sand (LB1816), Muddy sand (LB1818) and Muddy gravel to Muddy sandy gravel (LB1830).

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 highly 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.

A new workbook format was introduced in Scheme Year 18, the aim of this was to standardise the way in which laboratories provided data. Over the four exercises most laboratories completed the forms correctly and LB1818 commented that they liked the new auto-filling spreadsheets. A few laboratories commented that the spreadsheet was not auto-filling correctly; however all of the problems encountered were due to individual mistakes where data had not been entered correctly.

One of the main issues with the workbook was with the laser replicates section. If following the NMBAQC methodology laser subsamples should be passed through a 1mm sieve before laser diffraction. Sieving records a particle using the two smallest dimensions, while the laser diffraction measures the particle equivalent to a sphere of volume measured. Therefore particles measured by laser diffraction are bigger than those measured by sieves (Mason, 2011). **Hence, any greater than 1mm particles detected by laser should be removed and the remaining data re-scaled to 100%.**

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 is under review for the PS exercises in Scheme Year 18, 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. A modified workbook has been designed for use in Scheme Year 18 to enable laboratories to provide data in a comparable format. This has been modified slightly for Year 19 to resolve any issues that have arisen. 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 past sixteen 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 after the exercise deadlines as practicable. Participants that submit significantly incorrect data are contacted immediately to ensure that in-house checks can be implemented to ensure future quality assurance. The PS40, PS41, PS42 and PS43 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. The z-score method also assumes that the majority of respondents are correct and raised genuine concerns regarding technique and method bias. In Scheme Year 18 (2011/12) z-score analysis was run alongside cluster analysis using Euclidean distance matrices. PS40 and PS41 tentatively examined using confidence intervals, this approach will be examined in more depth in Scheme Year 19

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