Particle Size Analysis Component Annual Report
Scheme Operation 2022/2023 (Year 29)

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PARTICLE SIZE COMPONENT ANNUAL REPORT FROM APEM Ltd

SCHEME OPERATION – 2022/23 (Year 29)

Linked Documents (hyperlinked in this report):

Particle Size Exercise Results – PS84

Particle Size Exercise Results –PS85

Particle Size Exercise Results –PS86

Particle Size Exercise Results –PS87

List of Tables and Figures:

1. Introduction

The NE Atlantic Marine Biological Analytical Quality Control (NMBAQC) scheme is a quality assurance scheme developed on behalf of the UK competent monitoring authorities (CMAs). Its principal aim is to provide assessment of marine biological data contributing to UK national or European monitoring programmes.

The scheme also aims to develop and promote best practice in relation to sampling and analysis procedures through a range of training exercises, workshops, and literature guides.

The scheme includes seven biological components, each with its own set of training exercises and/or assessment modules.

APEM Ltd has been the administrative contractor for the Particle Size component since 2014 (Scheme year 21).

The Particle Size (PS) component of the scheme comprises two modules:

- The PS Ring Test (PS)
- The PS – Own Sample (PS-OS)

The PS module followed the same format as 2021/22; a series of exercises involved the distribution of test materials to participating laboratories and the centralised examination of returned data and samples.

The PS-OS module, introduced in the 2014/15 Scheme year, followed the same logistical format as the previous year. Selected participant samples are re-analysed by the NMBAQC Scheme PSA contractor, and the results are compared. The Particle Size Own Sample module is a training/audit module, and the purpose of this module is to examine the accuracy of particle size analysis for participants' in-house samples.

Seventeen laboratories signed up to participate in the 2022/23 PS module exercises (PS84, PS85, PS86 and PS87); seven were government laboratories and ten were private consultancies. Eleven laboratories signed up to participate in the PS-OS module exercises (PS-OS25, PS-OS26 and PS-OS27); six were government laboratories and five were private consultancies. One government laboratory had seven Lab Codes to submit 21 PS-OS samples for AQC analysis.

To reduce potential errors and simplify administration, Lab Codes were assigned with a prefix to determine the Scheme component; all codes for the Particle Size component were prefixed with "PSA_".

As in previous years, some laboratories elected to be involved in limited aspects of the Scheme. Competent monitoring authorities (CMAs) completing PSA in support of biological analysis for monitoring programmes (including in assessment of MPA (Marine Protected Areas), as evidence under MSFD (Marine strategy framework directive) and WFD (Water Framework Directive), as well as the CSEMP (Clean Seas Environmental Monitoring programme), must participate in this component of the Scheme. The Scheme is aware of other PSA methodologies (*e.g*. those used in the Regional Seabed Monitoring Plan) and encourages those involved in any relevant PSA monitoring programmes to participate in this Scheme, especially where pass/fail criteria can be used to assess overlapping aspects of different methodologies.

1.1 Assessing Performance

For 2022/23 (Scheme Year 29) both the PS and PS-OS reports followed a similar format, with each sample analysis section broken down for review, including sieve processing, laser processing and final data. Laboratories received a "Good" or "Review" flag based on their results; "Review" flags had accompanying comments as to where errors have been made and how to correct them. Review flags could be upgraded to « GOOD – following remedial action » provided the participant supplied evidence of completing required actions or re-submitted results within a month of the issue of the interim report.

1.2 Statement of Performance

Each participating laboratory received a copy of the interim results for each exercise; these included a summary of results provided by each laboratory and a basic discussion of any major outliers. Once any remedial actions, re-submissions and minor data changes were completed by participants a final version of each report was made available on the Scheme website. Further details and analysis can be found in this report.

At the end of the Scheme year each laboratory received a 'Statement of Performance' document (SoP), which included a summary of results for each of the Scheme's 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 two 2022/23 year PSA modules, PS and PS-OS are described in more detail below. A brief outline of the information 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.

2.2 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.

2.3 Data returns

Spreadsheet based workbooks for each circulation were distributed to participating laboratories via email and data returned to APEM Ltd via the NMBAQC Scheme email address. 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.

2.4 Confidentiality

To preserve the confidentiality of participating laboratories, each was identified by a four-digit Laboratory Code prefixed with "PSA_", to identify the scheme component. In October 2022 each participant was given a confidential, randomly assigned 2022/23 (Scheme year twenty-nine) Lab Code. Codes are prefixed with the Scheme year to reduce the possibility of obsolete codes being used inadvertently by laboratories, *e.g.* Laboratory number twelve in Scheme year twenty-nine (2022/23) was recorded as PSA_2912.

3. Particle Size Analysis (PS) Module

3.1 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 (PS84 and PS87), one mixed (PS85) and one coarse (PS86) were distributed in 2022/23. The samples were distributed in two stages; the first circulation (PS84 and PS85) was sent to participants on 31st October 2022 and the second circulation (PS86 and PS87) was sent on the 13th January 2023. For each circulation participants were given approximately 6 weeks to complete their analysis and send completed workbooks via email to APEM Ltd. PS84 and PS87 were derived from natural marine sediments; PS85 and PS86 replicates were prepared from a combination of natural sediments and artificially prepared commercial sand and aggregates; they were prepared at APEM's Letchworth laboratory as described below.

3.1.1 Asbestos testing

Following participant concerns raised during Scheme Year 25 (2018/2019) about the possible presence of asbestos in natural sediments used to create the PS exercises, all the natural sediments are now sent for asbestos testing prior to the creation of the samples. Sediments are only used when they have tested negative for asbestos; any that test positive are disposed of either in a landfill that has a specific permit authorising it to accept asbestos or in a non-hazardous waste landfill, provided it is self-contained. Details and results of asbestos testing can be requested by emailing APEM's NMBAQC email address.

3.1.2 Preparation of the Samples

The first PS circulation, PS84, was a sandy mud collected from natural marine environments from the Celtic Sea by the JNCC. Approximately 10 litres of visually similar sediment were collected and returned to the laboratory where it was wet sieved at 1.0mm to remove any particles larger than 1.0mm. Sediment that passed through the 1.0mm sieve was retained in a large tray, mixed and left to settle; excess water was removed before it was cored into replicate samples of approximately 200 grams in weight. The second exercise, PS85, was a mixed sample; known amounts of commercially acquired tropical reef aquarium gravel (split into half-phi intervals by dry sieving using a mechanical sieve shaker) was mixed with muddy sand collected from The Wash which had been pre-sieved through a 1mm sieve.

The third exercise, PS86, was created from known amounts of commercially acquired tropical reef gravel (split into half-phi intervals by dry sieving using a mechanical sieve shaker) mixed with coarse sand from the West of Orkney. The final exercise (PS87) was a created using known quantities of West of Orkney sand mixed with sandy mud from the Wash.

Five replicate samples from each of these exercises were sent to Kenneth Pye Associates Ltd (KPAL) for particle size analysis to assess the degree of inter-sample variation and to produce benchmark data. Where laser diffraction analysis was required, these replicates were analysed using a Beckman Coulter LS13320 laser diffraction instrument. The remaining replicates were randomly assigned to participating laboratories and distributed according to the Scheme timetable. Spare replicates were kept at the APEM Ltd. Letchworth laboratory in case of problems such as damaged samples during delivery or significant processing errors.

3.1.3 Analysis required

The participating laboratories were required to conduct particle size analysis on the samples following the NMBAQC Scheme's best practice guidance for particle size analysis to support biological data (NMBAQC Best Practice Guidelines (Mason, 2022)), either in-house or using a subcontractor. A summary of the sample as a written description of the sediment characteristics was to be recorded, with a qualitative visual assessment made prior to processing, using the Folk (1954) textural classification. In addition, the percentages of gravel, sand and silt/clay and any use of peroxide treatment or chemical dispersant were to be noted. Also requested was a breakdown of the particle size distribution, expressed as a weight or volume percentage at half-phi (ϕ) intervals, for each of the raw sieve data (>1mm), the raw laser data (<1mm) and the final merged dataset.

The 2022/23 workbooks followed the format of the updated 2021/22 workbooks to help enable the continuity of data through the workbook. All sieve and laser data are now entered into a single worksheet, with conditional formatting to flag up red cells to indicate possible data entry errors. Data provided in the "Laser Metadata" spreadsheet tab were for analytical purposes only and were not published in the Interim Results reports. Benchmark metadata were included in each sample report for participants to see how the Benchmark Lab analysed each sample.

Approximately seven weeks were allowed for the analysis of the first pair of PS samples sent out (PS84 & PS85) and approximately nine weeks for the second pair (PS86 & PS87).

3.2 Results

3.2.1 General comments

Seventeen laboratories subscribed to the exercises in 2022/23. For the first circulation (PS84 and PS85) thirteen subscribing participants provided results on time; PSA_2907 communicated that they would not be able to meet the December deadline but would submit data after the Christmas break; PSA_2908 communicated that they currently had no functional labs and they were in the process of acquiring new lab space so would participate once set up. PSA_2906 and PSA_2913 did not submit data and did not provide any further communication indicating non-participation. For the second circulation (PS86 and PS87) eleven participants provided results on time. PSA_2902 and PSA_2907 requested an extension to the submission deadline. PSA_2908 communicated non-participation due to lack of available lab space and PSA_2911 communicated that they would not be participating in this round due to the coarse nature of the samples. PSA_2912 submitted two results for PS87 as they are in the process of testing a new laser analyser. PSA_2906 and PSA_2913 did not submit data and did not provide any further communication indicating non-participation.

Most participating laboratories now provide data in the requested format, although some variations remain. As reported previously, 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. Detailed results for each exercise (PS84, PS85, PS86 and PS87) have been reported to the participating laboratories; additional comments are provided below.

3.2.2 Analysis of sample replicates (Benchmark Data)

Five replicate samples of the sediment used for the four PS distributions were analysed by KPAL to examine variability and establish benchmark data that participant results can be compared with. Replicate samples supplied by APEM were analysed, where required, using Endecotts British Standard 300mm and 200mm test sieves, Endecotts EFL 2000/2 and Retsch AS2001 Control 'g' sieve shakers and a Beckman Coulter LS13320 laser size analyser. In previous Scheme years replicates were analysed by both laser diffraction and sieve/pipette methods; however, as all participating laboratories are now conducting less than 1mm analysis by laser diffraction the testing of replicates for 2022/23 was undertaken only using a laser diffraction instrument.

The analysis results for the benchmark replicates were assessed by APEM to analyse the variability between the replicates and to establish the reproducibility of the samples. The analysis showed an overview of the sample including percentage Gravel, Sand and Mud along with a description of the sediment using the textural group from a Gradistat (Blott & Pye, 2001) output of the final data, e.g. Slightly Gravelly Muddy Sand. The processing of the sample was split into sieve and laser analysis.

Sieve analysis is displayed in a table with the raw weight recorded in each half phi interval from -6.5 to 0.0phi and the weight of the less than 1mm oven dried sample plus any sediment from the base pan of the sieve shaker. The percentage weight in each half-phi category is also displayed graphically in a bar chart for visual comparison.

Laser analysis included a table of the final laser data for each replicate with Gradistat outputs along with a graph showing the differential and cumulative percentage. The triplicate analysis undertaken to obtain the final laser data was presented in a table in Appendix 1. For each replicate sample the Coefficient of Variation (CV) was calculated for the D_{10}, D_{50} and D_{90} particle size in microns. The CV is most commonly expressed as the standard deviation as a percentage of the mean and describes the dispersion of a variable in a way that does not depend on the variables' measurement units. A low CV indicates a smaller amount of dispersion in the variable. BS ISO 13320 states that good laser reproducibility is shown for replicates when the %CV is <3% for the D_{50} and <5% for the D_{10} and D_{90}, all limits are doubled when the D_{50} was less than 10µm. In reality 3% and 5% are low and greater variability is expected in natural sediment samples therefore a maximum of 20% will be used as guidance.

Benchmark analysis of the replicates for Sample PS84 indicated an average composition of 60.33% sand and 39.67% mud, classified as 'Muddy Sand' according to the Blott & Pye (2012) scheme. Analysis of the triplicate laser analysis for each replicate sample showed that the %CVs for the D_{10}, D_{50} and D_{90} were well within the acceptable limits and therefore the replicates were deemed to have good reproducibility. Results for the individual replicates are provided in Tables 1, 2, 3, 4 and 5, and are displayed in Figures 1, 2 and 3 in the PS84 Report.

Sample PS85 was a mixed sediment and contained an average of 44.88% gravel, 52.19% sand and 2.92% mud, classified as a 'Sandy gravel' according to the Blott & Pye (2012) scheme. The replicates were analysed by dry sieving and laser analysis. The sieve data shows consistent results between the replicates and triplicate laser analysis showed extremely low variation, with %CV well below acceptable levels for each statistic. Results for the individual replicates are provided in Tables 1, 2, 3, 4 and 5, and are displayed in Figures 1, 2 and 3 in the PS85 Report.

Sample PS86 was a coarse sample and contained an average of 46.42% gravel, 52.94% sand and 0.61% mud, classified as a 'Sandy gravel' according to the Blott & Pye (2012) scheme. The replicates were analysed by dry sieving and laser analysis. The benchmark laboratory commented that it was not possible to take a laser subsample from the bulk before wet separation due to there being proportionally very little sediment <1 mm. Therefore, the sediment was wet separated at 1 mm, the >1 mm and <1 mm fractions dried, sieved, and weighed. Laser analysis was then performed on the <1 mm wet separated sediment. The sieve data shows consistent results between the replicates and triplicate laser analysis showed low variation. The %CV slightly high for the D_{10} of samples PSA_2938 (BM Rep3) and PSA_2940 (BM Rep 5) but below the acceptable levels for all other statistics. Results for the individual replicates are provided in Tables 1, 2, 3, 4 and 5, and are displayed in Figures 1, 2 and 3 in the PS86 Report.

Sample PS87 was a sandy sediment sample containing an average of 0.00% gravel, 97.87% sand and 2.13% mud, classified as 'Sand' according to the Blott & Pye (2012) scheme. Analysis of the triplicate laser analysis for each replicate sample showed that the %CVs for the D_{10}, D_{50} and D_{90} were well within the acceptable limits and therefore the replicates were deemed to have good reproducibility. Results for the individual replicates are provided in Tables 1, 2, 3, 4 and 5, and are displayed in Figures 1, 2 and 3 in the PS87 Report.

3.2.3 Results from participating laboratories

In each of the PS84, PS85, PS86 and PS87 reports, data provided by the participants are displayed in a series of tables and figures for comparison with each other and with the Benchmark Data. The Participant section provides three tables of data, the first outlining an overview of summary data including equipment and methodology used, the use of any chemical dispersants or pre-treatments, the percentage gravel, sand, and silt/clay recorded as well as the participants' post-analysis sediment descriptions. The second table provides the raw sieve weights for each half-phi interval submitted by each participant including the less than 1mm weights for the sieve shaker base pan fraction and the wet-separated and oven dried fraction and in the third table the final laser data submitted by each participant is shown. Tables showing the results of the triplicate laser analysis supplied and the Coefficient of Variance of the D_{10}, D_{50} and D_{90} are available in Appendix 3. These tables are accompanied by a series of graphs and bar charts which allow the results to be visually compared. Appendix 2 shows the data used to create the percentage gravel, sand, silt, and clay bar-charts. The final merged data submitted by each participant and the benchmark laboratory are provided in Appendix 4.

3.2.3.1 Eighty-fourth distribution – PS84

There was generally good agreement for PS84 between the results for the Benchmark replicates and those supplied by the participating laboratories (see Figure 1). Four participants (PSA_2902, PSA_2903, PSA_2910 and (PSA_2911) re-submitted or adjusted their data after the issue of the Interim Report. Participants PSA_2902 and PSA-2905 returned their samples to be re-analysed by the Benchmark lab. Table 6 in the report shows that none of the participants used chemical dispersants or peroxide pre-treatments. All participants recorded a Gradistat textural group of 'Muddy Sand' except for PSA_2905 who recorded it as 'Sandy Mud'. No gravel was recorded by any of the participants; percentage sand ranged from 47.44% (PSA_2905) to 69.59% (PSA_2910) and conversely the percentage of mud ranged from 30.41% (PSA_2910) to 52.56% (PSA_2905).

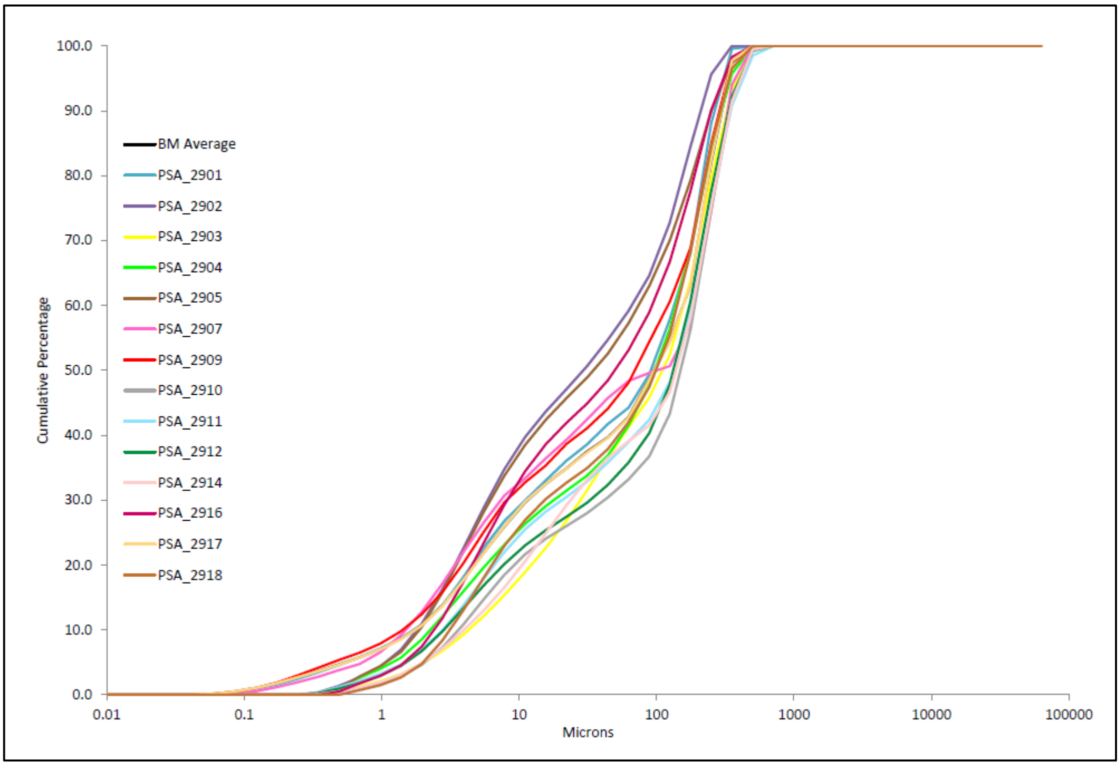


Figure 1. Particle size distribution curves for sediment distributed as PS84 (Figure 6 in PS84 Report).

As recorded in Table 6 of the PS84 report, all but three laboratories (PSA_2905, PSA_2911 and PSA_2914) followed the NMBAQC methodology. All participants analysed the sample using a laser analyser. Table 7 shows that three participants (PSA_2904, PSA_2910 and PSA_2912) also undertook sieve analysis. PSA_2910's data showed that any material that had been retained greater than 1mm during the wet split process passed through into the base pan during sieve analysis. PSA_2904 recorded 0.0057g greater than 1mm whilst PSA_2912 recorded 0.009g greater than 1mm.

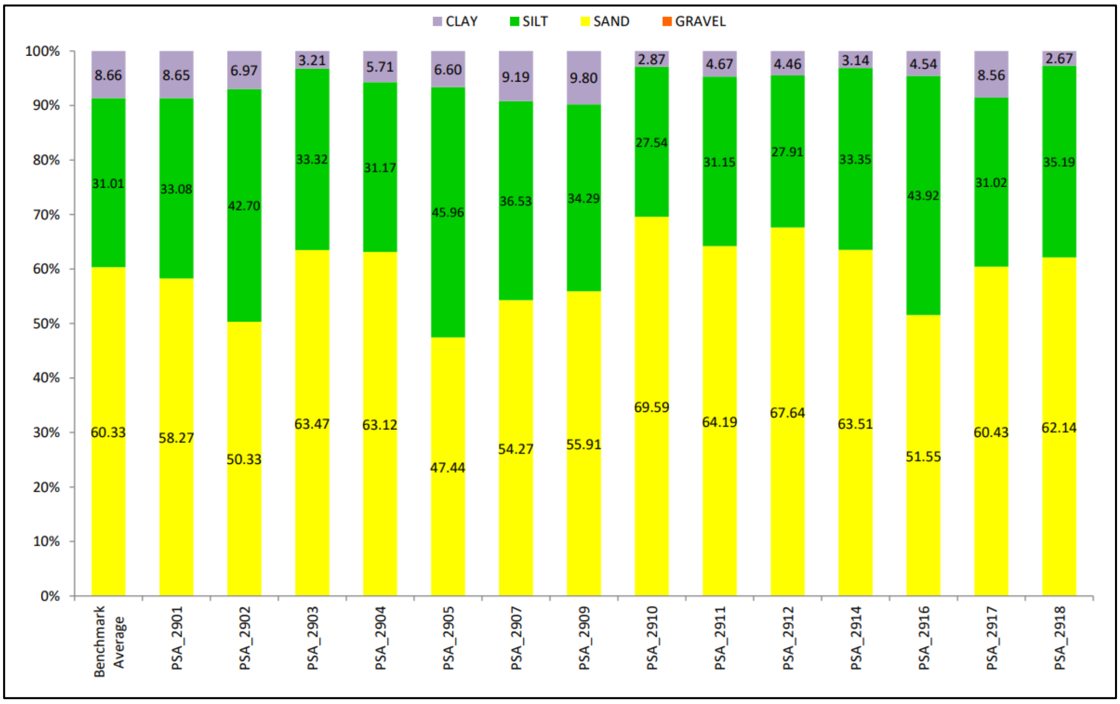


Figure 2. Stacked column chart showing the percentage gravel, sand, silt, and clay for sediment distributed as PS84 (Figure 7 in PS84 Report).

The sample showed some variation in the amount of clay recorded in relation to the model of laser analyser used. Those participants using Beckman Coulter instruments recorded a higher percentage of clay than those using Malvern Mastersizer instruments, as shown in Figure 2. Participants PSA_2901, PSA_2909, PSA_2917 as well as the Benchmark Lab use the Beckman Coulter LS13320 which uses a PIDS (Polarization Intensity Diffraction Scattering) system at the finer end, rather than diffraction, so provides better sensitivity than the Malvern system which employs diffraction of two different wavelengths of light (red and blue). Participant PSA_2907 is the only laboratory to use a Fritsch laser analyser, which recorded an amount of clay consistent with laboratories using the Beckman Coulter instruments.

3.2.3.2 Eighty-fifth distribution – PS85

There was good agreement for PS85 between the results from the analysis of the benchmark replicates and those from the participating laboratories (see Figure 3). Participant PSA_2903 was asked to review their result after the Interim Report due to an increased percentage gravel. However, it was found that there was an error in the replicate creation and the sample contained more greater than 1mm sediment than other samples. Participant PSA_2911's data in the Interim Report was displaced by 1 phi this was corrected and re-submitted. All participants had a Gradistat textural group of 'Sandy Gravel'. Participant PSA_2911 and

PSA_2914 do not follow the NMBAQC methodology and only sieve at 1-phi intervals above 1mm. Therefore, they are not assessed on the sieve aspect of the module. Excluding PSA_2903 and the two participants who don't undertake sieve analysis in the same way (PSA_2911 and PSA_2914), percentage of gravel (see Figure 4), ranged from 43.45% (PSA_2909) to 45.59% (PSA_2916). The percentage of sand ranged from 52.05% (PSA_2904) to 54.9% (PSA_2918) and the percentage of mud ranged from 0.77% (PSA_2910) to 3.53% (PSA_2909).

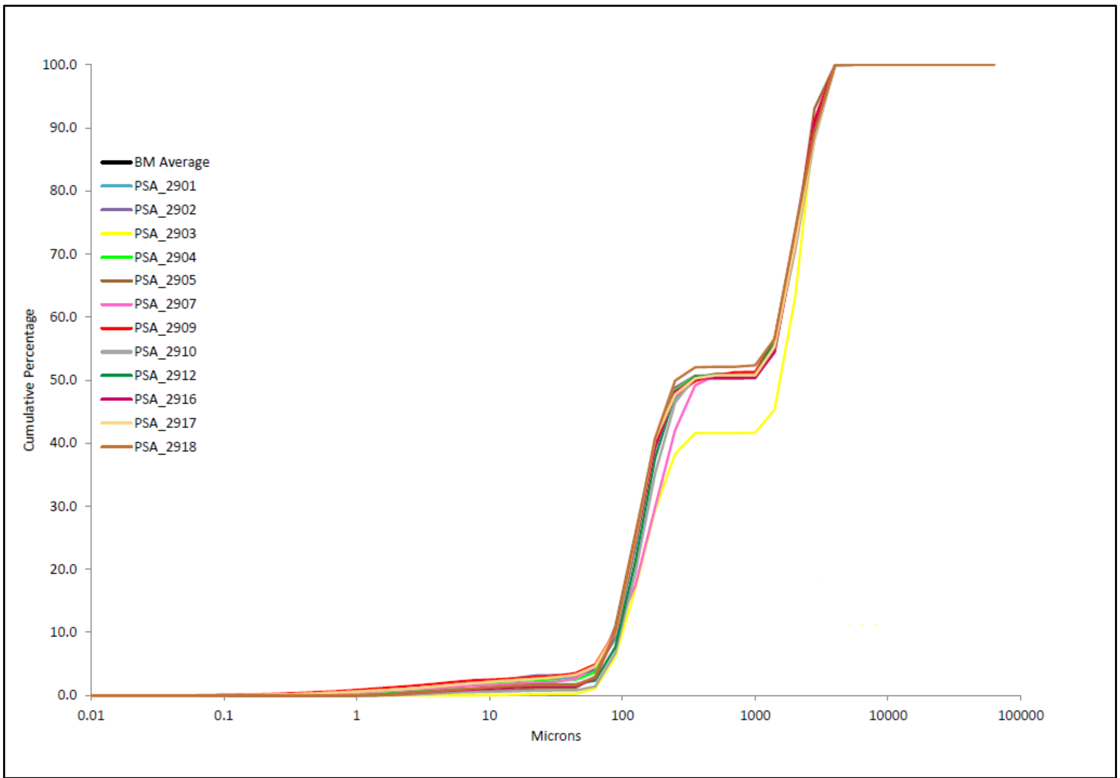


Figure 3. Particle size distribution curves for sediment distributed as PS85.

Despite the low quantities of mud present in PS85, Appendix 2 shows that as with PS84, the percentage of clay recorded showed some variation with laser manufacturer. The three participants using Beckman Coulter instruments recorded between 0.71% and 1.09% clay, whereas those using the Malvern Mastersizer instruments recorded between 0.00% and 0.38% clay. The Fritsch laser analyser was similar to the Beckman Coulter with a result of 0.62%.

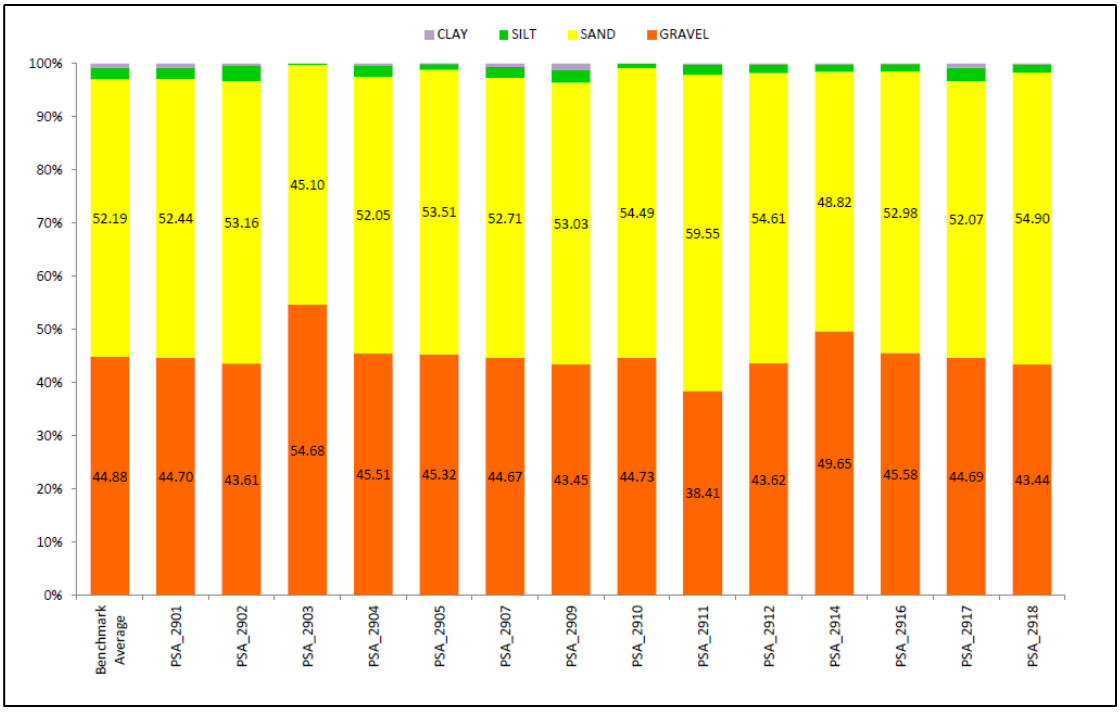


Figure 4. Stacked column chart showing the percentage gravel, sand, silt, and clay for sediment distributed as PS85 (Figure 7 in PS85 Report).

3.2.3.3 Eighty-sixth distribution – PS86

There was good agreement for PS86 between the results reported by the participating laboratories and those obtained for the benchmark replicates, as seen in Figure 5. The main issue with PS86 was recording the less than 1mm fraction; eight participants undertook laser analysis with the remaining five only undertaking sieve analysis. Of those choosing only to sieve, two labs (PSA_2916 and PSA_2918) added the less than 1mm fraction into the 710µm - 1000µm bin in the final data. Two participants (PSA_2904 and PSA_2910) chose to omit the less than 1mm fraction from the final merged data and participant PSA_2907 added the sediment less than 1mm to the 100 – 1400interval. These participants chose to re-submit their data following the interim report to achieve a “Good – following remedial action” flag.

All participants recorded a Gradistat textural group of ‘Sandy Gravel’.

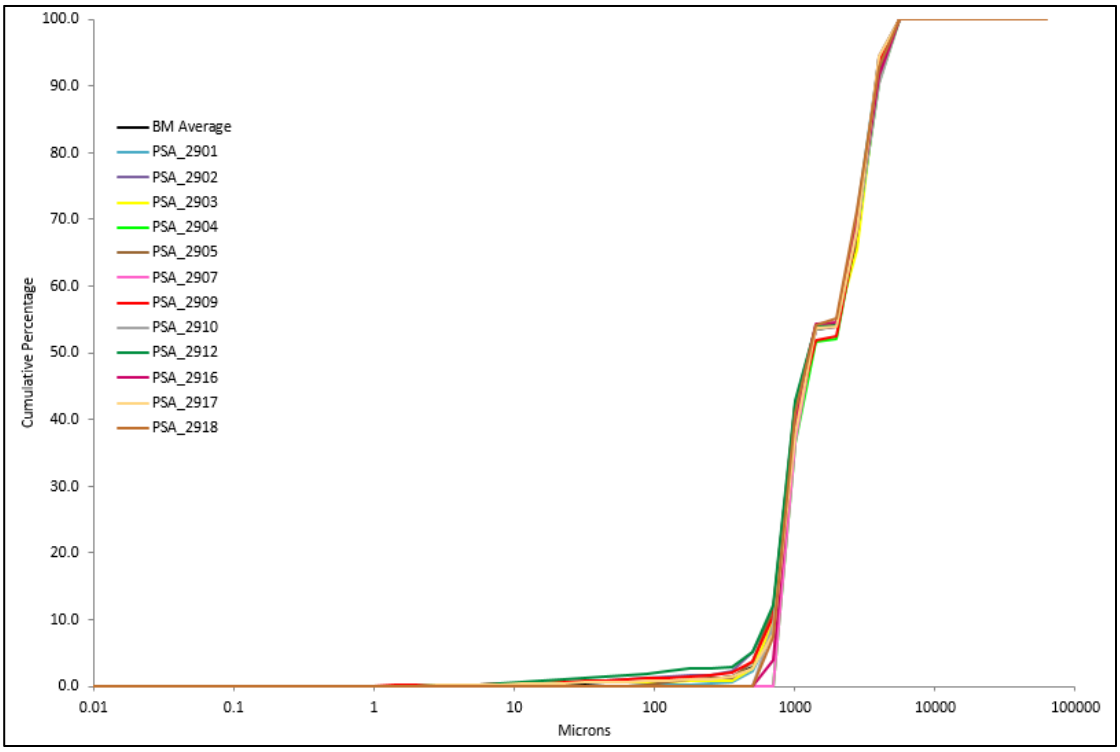


Figure 5. Particle size distribution curves for sediment distributed as PS86 (Figure 6 in PS86).

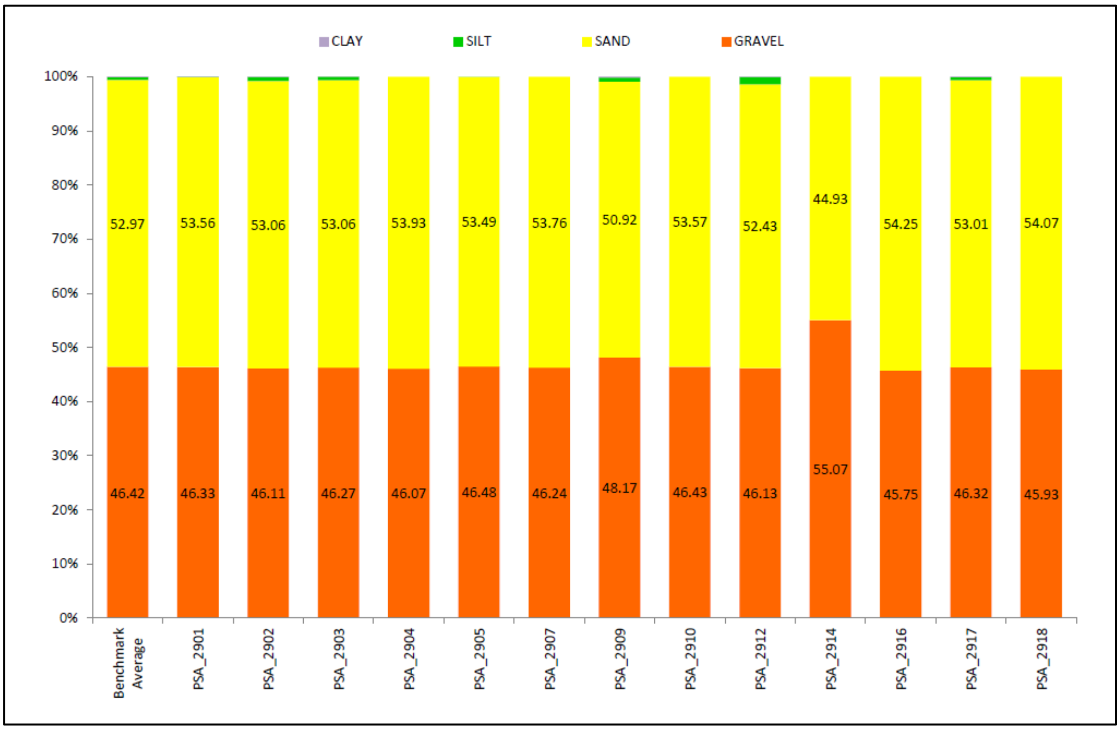


Figure 6. Stacked column chart showing the percentage gravel, sand, silt, and clay for sediment distributed as PS86 (Figure 7 in PS86 Report).

3.2.3.4 Eighty-seventh distribution – PS87

There was generally good agreement in results between the laboratories and the benchmark data (see Figure 7). At the time of writing participant PSA_2907 was still undergoing a review of their replicate and therefore have been omitted from the PS87 report for now. An updated version of the report may be published following the conclusion of the investigation. PSA_2912 submitted two sets of laser data as they were trialling a new laser analyser; PSA_2912 was undertaken using a Malvern Mastersizer 2000 and PSA_2912B was undertaken using a Malvern Mastersizer 3000. All participants had a Gradistat textural group of 'Sand'. The percentage of sand ranged from 97.10% (PSA_2902) to 100.00% (PSA_2903, PSA_2905, PSA_2912) and the percentage mud ranged from 0.00% (PSA_2903, PSA_2905, PSA_2912) to 2.90% (PSA_2902).

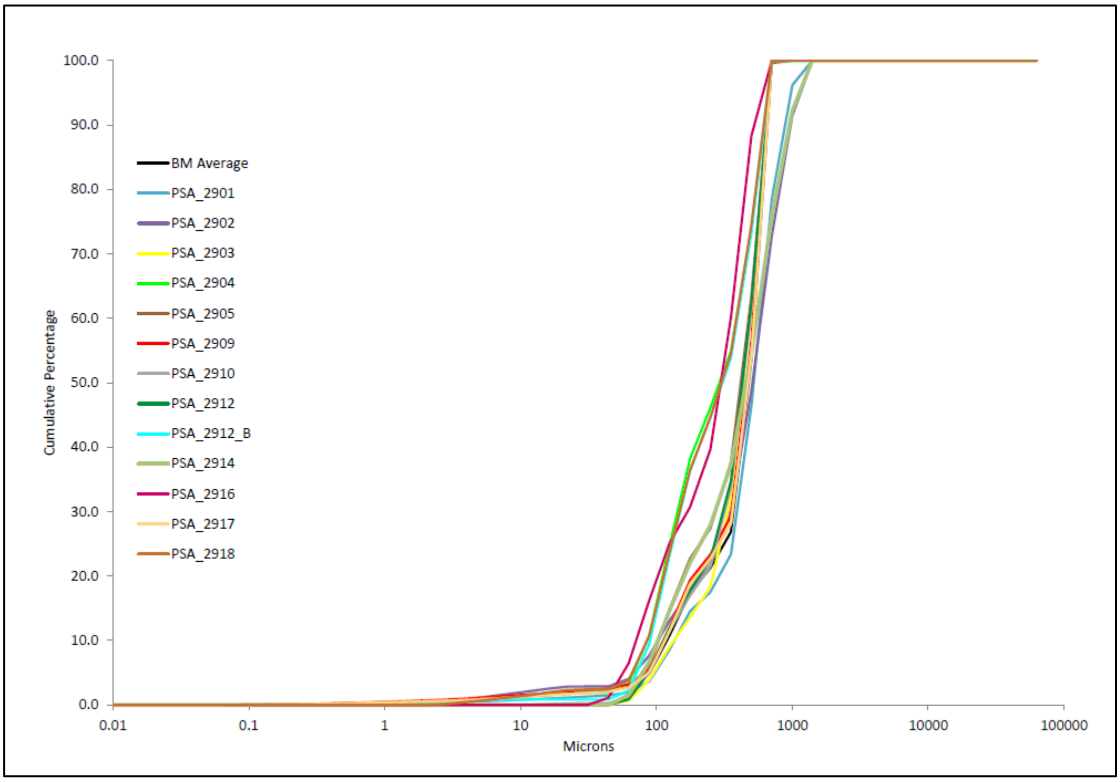


Figure 7. Particle size distribution curves for sediment distributed as PS87 (Figure 6 in PS87 Report).

Those that undertook sieve analysis (PSA_2904, PSA_2905, PSA_2909, PSA_2912 and PSA_2918) found small amounts (0.01g – 0.63g) of sediment greater than 1mm. The NMBAQC guidance states in "5.4.2 *Laser diffraction analysis of <1mm sediment fraction*" that "…if no sediment >1mm is left on the 1mm mesh [when preparing a laser sub-sample from the bulk], then no further analysis is required". With such small amounts of sediment greater than 1mm found in the entire sample it is unlikely that significant amounts of sediment greater than 1mm were present on the mesh when preparing a laser sub-sample and therefore sieve analysis did not have to be undertaken. Participants were not penalised for undertaking this extra analysis as it had little effect on the overall distribution of the sample.

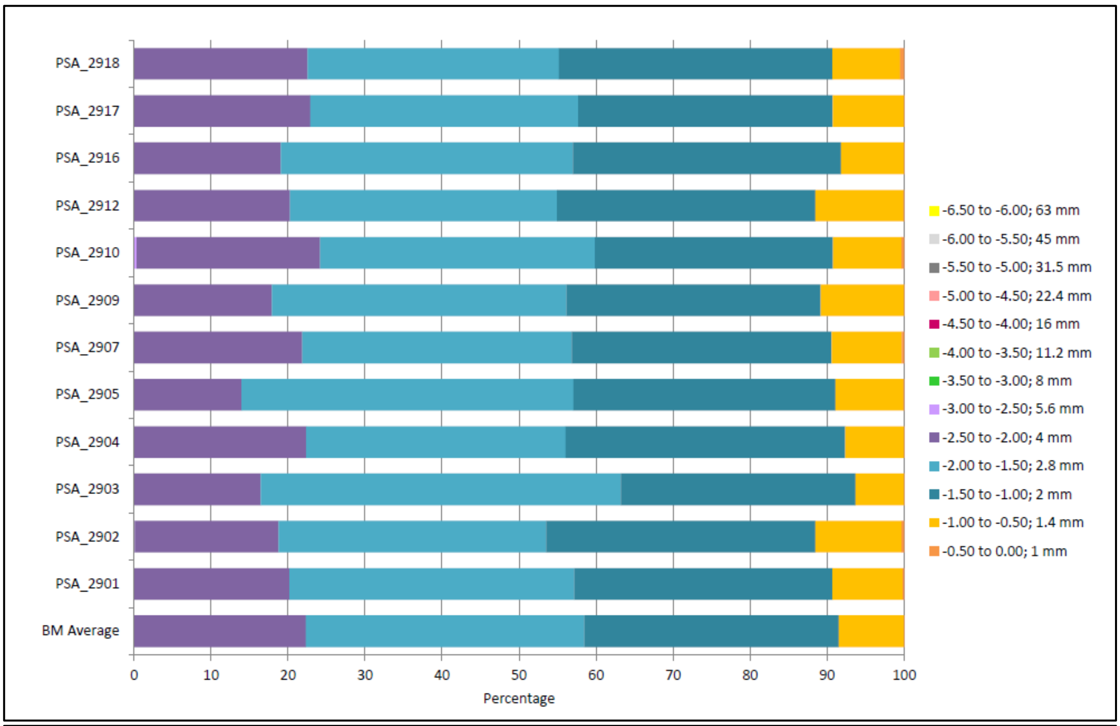
Figure 8. Stacked column chart showing the percentage gravel, sand, silt, and clay for sediment distributed as PS87 (Figure 7 in PS87 Report).

3.2.4 Discussion

The exercise reports show that most participants follow the NMBAQC methodology for these exercises; those that do not, do so for genuine reasons. PSA_2911 do not undertake analysis of sediment greater than 1mm so chose to only participate in the laser analysis for PS84 and PS85.

3.2.4.1 Sieve Analysis (>1mm)

The two exercises that contained larger quantities of sediment greater than 1mm (PS85 and PS86) show that the dry sieve analysis (>1mm) undertaken by participants was generally in agreement with each other and the benchmark data (see Figure 9). There is some variation, but this is to be expected with varying sieve times and amplitudes. The benchmark lab recorded a sieve time of 10mins and amplitude of 1.5mm/'g'. Of the sieve metadata provided by participants, sieve time varies from 2 to 30 minutes; sieve amplitude often didn't include a unit of measurement. Units of measurement may vary due to differing brands of sieve shaker. Sediment type may also be a contributing factor, brittle or chalky material may break up more easily and the longer and more vigorously the sample is shaken the greater the effect will be on the sample.



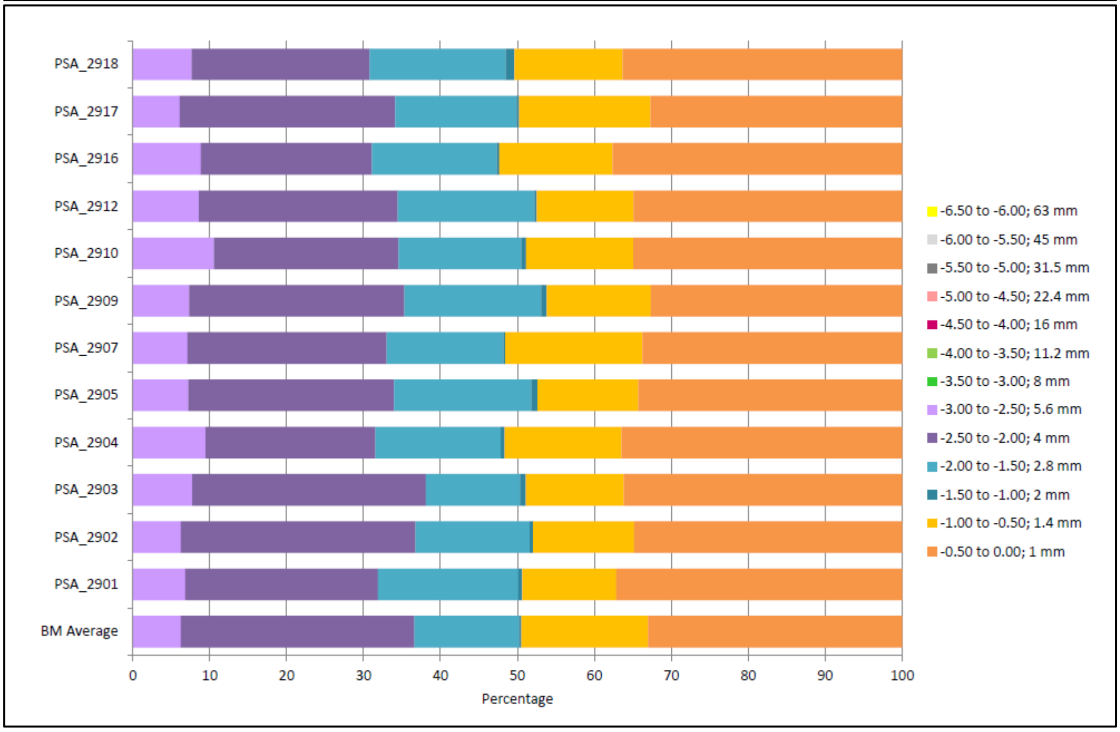


Figure 9. Bar charts showing raw sieve data as percentages in each half-phi interval for PS85 and PS86

3.2.4.2 Laser Analysis (<1mm)

Figure 10 shows the cumulative and differential curves for the laser data for each exercise. Although the results continue to show improvement from previous years, laser analysis remains the main source of variability between participants. All participants re-scaled their laser data to 100% before merging with the sieve data; where the final laser data provided included sediment >1mm, data were re-scaled to include only the <1mm fractions for comparisons with benchmark and other participant data.

As in previous years it was apparent in the exercises that required laser analysis and had a significant mud fraction (PS84) that there were some differences in results depending on which laser instrument was being used. The participants using the Beckman Coulter and Fritsch instruments recorded a higher percentage of clay than those using Malvern instruments, the Beckman Coulter instruments have greater measurement sensitivity and along with the Fritsch analyser were the only instruments capable of detecting particles below 0.345µm. The results obtained using the Beckman Coulter instruments also showed a much greater degree of similarity to each other than those using generated using the Malvern instruments. However, there were still slight differences detected between the participants using Coulter instruments, which could be due to differences in the samples supplied to each lab, different sub-sampling, sample dispersion and/or sample presentation procedures being used.

These differences between laser manufacturers were taken into consideration when comparing participant data with the Benchmark data especially where participants used the Malvern analysers as the Benchmark data is created using a Beckman Coulter.

Laser metadata are very important in helping to identify where possible mistakes are made and whether it is an issue with the laser or a sample preparation problem. For this reason, provision of metadata is a compulsory requirement. The majority of participants supplied laser metadata in the current year, PSA_2905 were the only participant to provide no laser metadata beyond the laser model and dispersion unit for any of the exercises.

The NMBAQC recommends using the Mie Theory model, a Particle Refractive Index of 1.55 and a Particle Absorption Index of 0.1, the dispersant used is water which has a Refractive Index of 1.33. Based on the information supplied, most participants are now using the NMBAQC Guidance recommendations. Participants that were not following the recommendations were reminded to do so in their results.

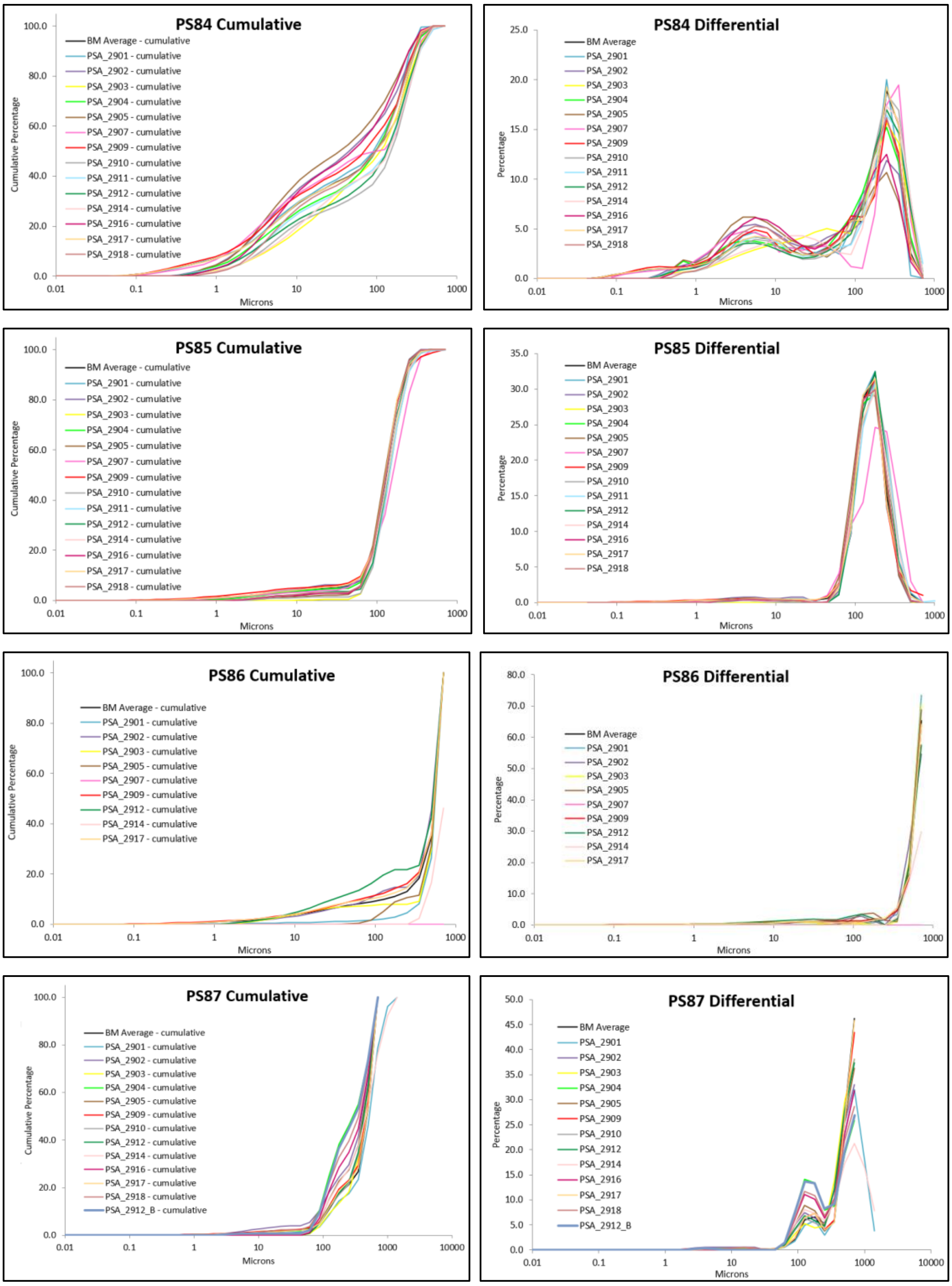


Figure 10. Cumulative and differential final laser data provided by participants for exercises PS84, PS85, PS86 and PS87.

For Exercises PS84, PS85, PS86 and PS87 all the participants that submitted metadata are now using the Mie Theory analysis model. All of the participants that provided metadata information used a Particle Absorption Index of 0.1. Most participants used a Particle Refractive Index of 1.55 while three participants used a Particle Refractive Index of 1.52

(PSA_2903, PSA_2904 and PSA_2911). All participants using Beckman Coulter laser analysers used the PIDS (Polarized Intensity Differential Scattering) system as the fines extension; all participants using Malvern Mastersizer instruments used both the red and blue light wavelengths.

There remains a degree of variation in the pump and stirrer speeds and the use of ultrasonics, this could potentially be standardised in future scheme years.

These factors are probably mostly responsible for the variation in the laser size distributions seen in Figure 9. It is not always obvious why a result appears to be different without detailed laser metadata. In addition to laser instrument set-up conditions and performance there are other factors that could be affecting the results, including sample preparation, sample dispersion methods and sample presentation to the laser instrument, about which little or no information has been provided.

3.2.5 Application of NMBAQC Scheme Standards and Laboratory Performance

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) and Water Framework Directive (WFD) monitoring programmes. With this aim, performance target standards were defined for certain Scheme modules and applied in 1996/97 (Scheme year three). 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. An overall summary of the data reported by each participant is presented in each of the PS exercise reports, and along with this each participant receives a results table outlining their individual performance. 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 still under review for the PS exercises, in 2021/22 (Scheme year 28) a “Good” or “Review” flag has been issued for Sieve analysis (>1mm), Laser Analysis (<1mm) and Final Data. This aims to highlight any potential errors but will not be used to assess the performance of a laboratory. As this is a training exercise rather than a proficiency test, participants are encouraged to review their results especially where “Review” flags have been issued and can re-submit improved data after the issue of the interim report. Each laboratory was issued with a Statement of Performance certificate outlining their results and participation in the Scheme.

4. Particle Size Own Sample Analysis (PS-OS) module

4.1 Description

The Particle Size Own Sample (PS-OS) module was first introduced in Scheme year 21 (2014/15) as a training/audit module and now is in its ninth year of operation. Participants' "own" samples are re-analysed by the NMBAQC Scheme PSA contractor and the results are compared. The purpose of this exercise is to examine the accuracy of particle size analysis for participants' in-house samples. In its first year (2014/15) the PS-OS exercises carried a trial Pass/Fail criterion based on the correlation between the participant data and the AQC data. After discussions between KPAL, APEM and the Scheme's PSA Contract Manager (Claire Mason, Cefas), it was decided that a more simplistic approach to analysing the results would be more appropriate in identifying errors in participants' results. The results now follow a similar format to the PS exercises and were split into sieve processing, laser processing and final data. Participants received a "Good" or "Review" flag based on their results. Where a "Review" flag was issued comments were supplied detailing problems that had arisen and where to find information to help address them.

4.1.1 Analysis required

Laboratories were requested to submit details of a survey with at least 12 samples from their previous year's Clean Seas Environment Monitoring Programme (formerly NMMP) samples, or similar alternative sampling programmes (if not responsible for CSEMP samples), along with the associated PSA data. Once these data were provided, three samples were randomly chosen by APEM Ltd to be re-analysed by the NMBAQC Scheme's PSA contractor.

Spreadsheet based workbooks were distributed to each participating laboratory via email for each PS-OS exercise. These were to be returned to APEM Ltd via the NMBAQC Scheme email address (nmbaqc@apemltd.co.uk).

In each workbook a written description of the sediment classification was to be recorded, a visual estimate was made prior to analysis and a post analysis classification based on the percentages of gravel, sand and silt/clay and the Folk (1954) terminology. Any use of hydrogen peroxide treatment or chemical dispersant was also to be recorded. Also requested was a breakdown of the particle size distribution of the sediment, expressed as a weight or weight percentage of sediment in half-phi (ϕ) intervals, as well as sieve and laser metadata to provide insight into laboratory procedures, especially for the laser analysis.

The different components of each PS-OS sample (< 1mm, > 1mm and laser sub-sample) were to be sent to APEM's Letchworth laboratory to be passed on to the NMBAQC Scheme PSA contractors. The two sets of results were then compared by APEM Ltd.

Following on from delays resulting from the Covid-19 pandemic the timetable for the PS-OS module was altered to allow participants more time to submit data and to speed up reporting of results to participants. Participants who took part in Batch 1 submitted data by 30th November 2022, had samples selected by the 23rd December 2022 and reports were issued at the beginning of April 2023; Batch 2 submitted data by 24th March 2023, had samples selected by the 28th April 2023 and reports were issued by the end of June 2023.

4.2 Results

4.2.1 General comments

Eleven laboratories subscribed to the PS-OS module in 2022/23. One of the eleven laboratories had seven lab-codes to facilitate multiple PS-OS submissions. Ten of the eleven laboratories that subscribed to the module provided data and samples for re-analysis. Participant PSA_2906 did not provide any data or communication of non-participation. Five participants submitted data in Batch 1 and five participants submitted data in Batch 2, including the lab with multiple LabCodes

Each laboratory received detailed comparisons of their data with the re-analysis results obtained by the NMBAQC Scheme's contractor. Where the original analysis was performed by the Scheme's contractor an external auditor was used to re-analyse the samples. Results were split into sieve processing, laser processing and final data. At the end of each report participants received a "Good" or "Review" flag based on their results; where "Review" flags were issued, comments were made on errors that had arisen and where possible information was provided to help resolve problems.

All the laboratories that provided samples provided all necessary fractions of their sample for re-analysis; except for participant PSA_2903 who provided a dried laser sub-samples and therefore this required re-wetting and mixing into a soft but stiff paste consistency in order to extract representative laser subsamples. Participant PSA_2902 bagged the sediment retained on each sieve in the original analysis separately. The AQC laboratory noted that this makes it difficult to empty 100% of the sample from the bags, potentially leading to slight underestimation of the sample weight, although every effort was made to extract as much sediment as possible. There is no need to bag each individual sieve fraction, particularly as this only serves to hamper rather than hinder any reanalysis.

There was generally good agreement between the participants and the AQC results, particularly in terms of basic sediment textural classification (see Table 1). Differences are highlighted in blue.

Table 1. Gradistat sediment descriptions from the primary data and the AQC re-analysis. Taken from Table 6 of the individual PS-OS reports.

Lab	Sample	Primary Sediment Description	AQC Sediment Description
PSA_2901	PS-OS 25	Muddy Sand	Muddy Sand
	PS-OS 26	Sandy Mud	Muddy Sand
	PS-OS 27	Gravelly Muddy Sand	Gravelly Muddy Sand
PSA_2902	PS-OS 25	Slightly Gravelly Sand	Slightly Gravelly Sand
	PS-OS 26	Gravelly Sand	Gravelly Sand
	PS-OS 27	Sandy Gravel	Sandy Gravel
PSA_2903	PS-OS 25	Muddy Sandy Gravel	Muddy Sandy Gravel
	PS-OS 26	Gravelly Muddy Sand	Gravelly Muddy Sand
	PS-OS 27	Gravelly Mud	Gravelly Mud
PSA_2904	PS-OS 25	Slightly Gravelly Muddy Sand	Slightly Gravelly Muddy Sand
	PS-OS 26	Slightly Gravelly Muddy Sand	Slightly Gravelly Muddy Sand
	PS-OS 27	Gravelly Muddy Sand	Gravelly Muddy Sand
PSA_2909	PS-OS 25	Sandy Gravel	Sandy Gravel
	PS-OS 26	Gravelly Mud	Gravelly Mud
	PS-OS 27	Gravelly Sand	Gravelly Sand
PSA_2916	PS-OS 25	Sand	Slightly Gravelly Sand
	PS-OS 26	Sand	Slightly Gravelly Sand
	PS-OS 27	Gravelly Muddy Sand	Gravelly Mud
PSA_2917	PS-OS 25	Slightly Gravelly Muddy Sand	Slightly Gravelly Muddy Sand
	PS-OS 26	Gravelly Muddy Sand	Gravelly Muddy Sand
	PS-OS 27	Slightly Gravelly Sand	Slightly Gravelly Sand
PSA_2918	PS-OS 25	Slightly Gravelly Sand	Slightly Gravelly Sand
	PS-OS 26	Gravely Sand	Gravely Sand
	PS-OS 27	Sandy Gravel	Sandy Gravel
PSA_2921	PS-OS 25	Muddy Sandy Gravel	Muddy Sandy Gravel
	PS-OS 26	Sand	Sand
	PS-OS 27	Slightly Gravelly Muddy Sandl	Slightly Gravelly Muddy Sandl

PSA_2922	PS-OS 25	Slightly Gravelly Sand	Slightly Gravelly Sand
	PS-OS 26	Slightly Gravelly Sandy Mud	Slightly Gravelly Muddy Sand
	PS-OS 27	Sand	Sand
PSA_2923	PS-OS 25	Slightly Gravelly Sand	Slightly Gravelly Sand
	PS-OS 26	Slightly Gravelly Muddy Sand	Slightly Gravelly Muddy Sandl
	PS-OS 27	Slightly Gravelly Muddy Sandl	Slightly Gravelly Muddy Sandl
PSA_2924	PS-OS 25	Sand	Sand
	PS-OS 26	Sandy Mud	Sandy Mud
	PS-OS 27	Mud	Mud
PSA_2925	PS-OS 25	Slightly Gravelly Muddy Sand	Slightly Gravelly Muddy Sand
	PS-OS 26	Sand	Sand
	PS-OS 27	Slightly Gravelly Sandy Mud	Slightly Gravelly Sandy Mud
PSA_2926	PS-OS 25	Slightly Gravelly Sand	Slightly Gravelly Sand
	PS-OS 26	Slightly Gravelly Sand	Slightly Gravelly Sand
	PS-OS 27	Muddy Sand	Slightly Gravelly Muddy Sand
PSA_2927	PS-OS 25	Muddy Sand	Slightly Gravelly Muddy Sand
	PS-OS 26	Sand	Slightly Gravelly Sand
	PS-OS 27	Sandy Mud	Slightly Gravelly Sandy Mud
PSA_2928	PS-OS 25	Slightly Gravelly Sand	Slightly Gravelly Sand
	PS-OS 26	Slightly Gravelly Sand	Slightly Gravelly Sand
	PS-OS 27	Slightly Gravelly Sand	Slightly Gravelly Sand

The differences in textural groups between participant PSA_2901 and the AQC data were due to very small differences that shifted the sediment descriptions. In sample PSA_2901 PS-OS 26 the AQC data recorded 1.19% less mud than the. This led to the AQC laboratory recording the sample as "Muddy Sand" and the primary analysis recording it as "Sandy Mud".

For samples PSA_2916 PS-OS 25 and 26, PSA_2926 PS-OS 27 and PSA_2927 PS-OS 25, 26 and 27, the AQC re-analysis recorded very small amounts of sediment greater than 1mm (0.03g, 0.06g and 0.01g for PSA_2927 PS-OS 25, 26 and 27 respectively; 0.02g for PSA_2926 PS-OS 27 and 0.01g for PSA_22916 PS-OS 25 and 26). The NMBAQC guidance states that "…if no sediment >1mm is left on the 1mm mesh [when preparing a laser sub-sample from the bulk], then no further analysis is required". With such small amounts of sediment greater than 1mm found in the entire sample it is unlikely that significant amounts of sediment greater than 1mm were present on the mesh when preparing a laser sub-sample and therefore sieve analysis did not have to be undertaken.

The greater than 1mm data created by dry sieving was in general very good, there were a few discrepancies, but these are to be expected due to factors such as breakage of particles during repeat analysis and variations in sieving time and vibration amplitude.

Small amounts of variability particularly in percentage clay shown in Figures 11, 12 and 13 can be explained by differing laser instruments used by the AQC lab and participants. As discussed earlier in this report, the Malvern Mastersizer 2000 and 3000 instruments do not have the same resolution as the Coulter LS13320, especially at the finer end; the Coulter uses a PIDS (Polarization Intensity Differential Scattering) system at the bottom end, rather than diffraction, so provides better sensitivity than the Malvern system which employs diffraction of two different wavelengths of light (red and blue). Often the Coulter system reports higher mud content than the Malvern machines and the distributions produced by the Malvern tend to be more smoothed, and less able to identify discrete size modes. The output size distribution from the Malvern instruments machines is very dependent on the diffraction pattern interpretation model used; this can be selected by the operator as "General Purpose, Unimodal, and Multimodal etc." and can give rise to uncertainty. There is no such specification requirement with the Coulter instruments.

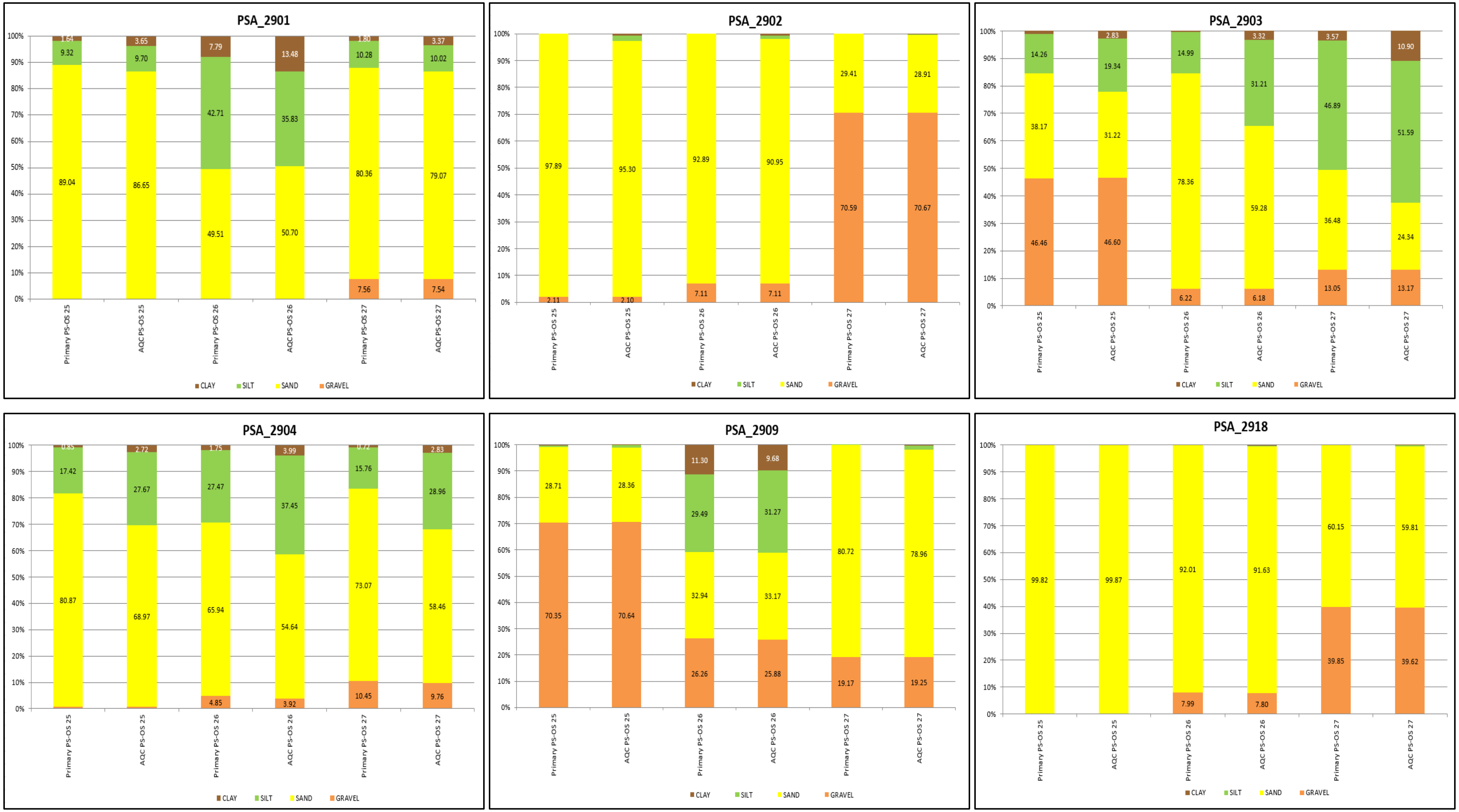


Figure 11. Bar charts showing percentage gravel, sand, silt, and clay in the PS-OS module from laboratories PSA_2901, PSA_2902, PSA_2903, PSA_2904, PSA_2909 and PSA_2918.

Figure 12. Bar charts showing percentage gravel, sand, silt, and clay in the PS-OS module from laboratories PSA_2916, PSA_2917, PSA_2921, PSA_2922, PSA_2923 and PSA_2924.

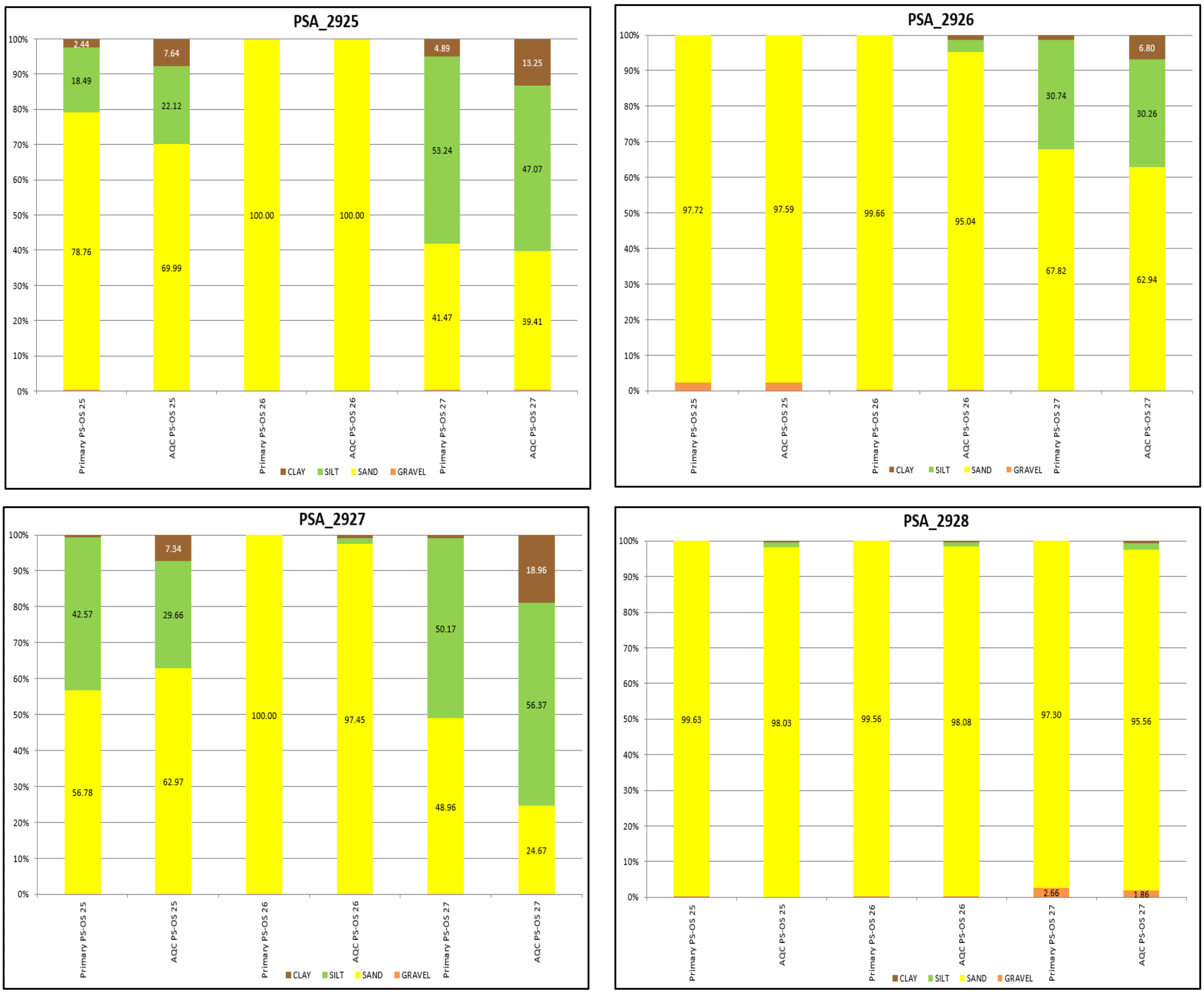


Figure 13. Bar charts showing percentage gravel, sand, silt, and clay in the PS-OS module from laboratories PSA_2925, PSA_2926, PSA_2927and PSA_2928.

4.3 Discussion

As in previous years, differences in laser analysis are still the main area of concern in the PS-OS samples. The interpretation of the methodology set out in the NMBAQC Best Practice Guidelines (Mason, 2022), in particular how the laser analysis is undertaken still appears to be a possible issue in some cases. These guidelines, originally written in 2011, were based on the widespread use at that time amongst participants of Malvern Instruments laser diffraction instruments that have 15 – 25 second standard run times and generally are restricted to the analysis of material < 1mm in size. The original methodology suggested that:

1. A homogenised sub-sample of approximately 100ml is taken from the bulk sample for laser analysis (Laser Pot).

2. A small representative sub-sample is taken from the Laser Pot and passed through a 1mm sieve using as little water as possible (Replicate 1).

3. All of Replicate 1 is then run through the laser at the desired obscuration, producing three run results.

Steps 2 and 3 are then repeated to create Replicates 2 and 3, giving a final result of 9 runs to create the final laser data, the average of these 9 runs.

The completion of nine analyses, and subsequent merging of results is necessarily a time-consuming process, especially if standard run times longer than 15 to 25 seconds are used (e.g. 60 seconds is standard with Beckman Coulter instruments (if the PIDS system is activated). It has been demonstrated by KPAL that, for most samples, there is little practical benefit in routinely carrying out analysis of three replicate sub-samples if samples are homogenised properly both before the laser sub-sample is taken from the bulk sample and when the test sample is taken from the laser sub-sample, and the sample is adequately dispersed prior to presentation to the instrument. In relatively rare instances where samples consist very largely of > 1mm size material and it is impractical to obtain a representative laser sub-sample from the bulk sample, more consistent laser results can be obtained by taking a laser sub-sample from the wet separated < 1mm fraction of the sediment, rather than from the bulk sample.

Where samples display, or are suspected of, unstable behaviour, such as time-dependent agglomeration, one or more repeat runs of the same test sample should be carried out, and additional replicate test samples analysed. Sometimes this may require repeat runs of more than three replicates to fully characterise agglomerative behaviour, and to establish the best dispersal procedures required to obtain repeatable results (e.g. ultrasonic treatment before as well as during the analysis run, and/or use of chemical dispersants). If the laser sub-sample is visually heterogeneous, and/or during the preparation of the test sample it is observed that small amounts of sand are present within a mainly muddy matrix, two or more test samples should be analysed. Additionally, for QA purposes, it is good practice to carry out at least duplicate analysis on 1 in 10 samples. The guidance has been updated to incorporate most of these findings and recommendations, with some further follow up expected at future NMBAQC PSA workshops. The most recent version of the guidance can be viewed in Mason (2022).

The returns for the 2021/22 PS-OS module showed that some laboratories, particularly those using Coulter instruments, in routine case work only run one laser test sample, with, for QA demonstration purposes, replicates run every 10th, 20th or 50th sample, dependent on sediment type (less frequently for well sorted uniform sand samples than for poorly sorted muddy sand and muddy sandy gravel mixtures). The results obtained by KPAL, for the NMBAQC replicates samples prepared by APEM since 2014/15, demonstrate that the high degree of repeatability which can be obtained when strict analysis protocols are followed, and that a high degree of confidence can be placed in the results obtained for any individual analysis.

The PS-OS module also revealed that a few participants do not follow the NMBAQC methodology for routine samples. This generally occurs when a participant does not have access to a laser analyser, in this case only the sieve and final data can be compared. Participants are encouraged to participate even when samples have been analysed following a different methodology as long as details of the methodology used are presented clearly. Although re-analysis will be undertaken following the NMBAQC methodology this gives a chance to compare how results differ when using alternate methodologies. Using a different methodology will always be taken into consideration when comparing the primary and AQC analysis.

5. Conclusions and Recommendations

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

1. Laboratories should ensure that they follow the NMBAQC methodology when participating in the Particle Size (PS) Ring Test. The PS Ring Test is designed to test whether all participants are getting comparable results when they follow the same methodology. It is therefore important that only the NMBAQC methodology (Mason, 2022) is used where possible and that results for 3 x 3 laser analyses are provided. Participants who do not have access to a laser analyser will be permitted to use alternate methods for samples that contain sediment less than 1mm as long as the method used is detailed in the summary section of the workbook. Participants can choose to opt out of either the sieve or laser aspects if they do not routinely undertake that type of analysis. The participant must let the administrator know at the start of the scheme year if they wish to opt out of any analysis. Results will only be provided for the analysis that was undertaken and a note will be put on the Statement of Performance that the participant has opted out of certain points.

   Samples for the PS-OS module can be analysed following alternative in-house methods however these must be thoroughly described and the participant should be aware that re-analysis will be undertaken following the NMBAQC methodology. Samples provided for PS-OS which have been routinely analysed do not necessarily have to provide 3 x 3 laser analysis data but should show that appropriate QC checks have been carried out, including on the final data set.

2. Participants should review their data prior to submission. Errors in datasets can often be spotted in the summary statistics, e.g. percentage gravel, sand and silt/clay, before the data are submitted. All parts of the workbook should be double checked before submission to ensure that they have all been completed correctly. This will help eradicate typing and transcription errors. The workbook was updated for the current Scheme Year (Year 28) to help enable the continuity of data through the workbook. Conditional formatting flags up red cells where there are possible data entry errors.

3. The current NMBAQC Scheme Pass/Fail criteria for the PS modules are under review. Currently results are broken down for review, including sieve processing, laser processing and final data. Laboratories then received a “Good” or “Review” flag based on their results; “Review” flags came with accompanying comments as to where mistakes have been made and how to correct them. This approach was thought to be more informative and would help participants to identify errors and correct any issues

for future exercises. Following the publication of 'Statistical comparisons of sediment particle size distributions' (Barry *et al*., 2021) in Continental Shelf Research, data from previous and future reports will trial this new statistical method of comparing the benchmark and participant data to understand if we can achieve a pass/ fail criteria for the particle size component, with the possibility of a report detailing the outcomes available in the next couple of scheme years.

4. **A Review is not a fail.** Although every attempt is made to ensure that all replicates are as similar as is humanly possibly there will naturally be some variation, particularly in natural mud samples. A review flag is just to point out that your analysis does differ from that of the Benchmark Lab and other participants. We encourage participants to review their data and if required request a new replicate or ask for their replicate to be re-analysed by the Benchmark Lab for a comparison.

5. **A two-day workshop was held at Cefas' Lowestoft laboratory in March 2023.** The first day was a practical session in the lab; a team from Meritics demonstrated the new Bettersizer laser analyser which has the capability to capture particle shape. Whilst staff from Cefas' sediment lab demonstrated an overview of methodology including sub-sampling and sample handling including mitigations for low levels of asbestos in samples. The second day was a series of presentations; Lydia McIntyre-Brown (APEM) gave an update on the NMBAQC Scheme, David Van Avermate (ILVO) looked at defining pass/fail criteria from ring test data for accreditation purposes, Claire Mason (Cefas) looked at the inclusion of cobbles in particle size data, Clement Garcia looked at the Marine Natural Capital Programme and it's relevance to PS methodology, Keith Cooper looked at the One Benthic project and Ken Pye and Simon Blott (KPAL) gave reflections on the NMBAQC PSA Scheme thus far.

6. **Health and Safety.** Recently the presence of asbestos in marine samples has been brought to light, although safe when the sample is wet, asbestos particles could become air-borne when analysing a particle size sample particularly during the dry sieving process. At the PSA workshop in December 2017, laboratories were informed how to mitigate the hazards associated with analysing samples that may contain asbestos and at the March 2023 workshop these mitigations were demonstrated. All the natural material used to create PS ring test samples continues to be sent for presence/absence of asbestos before being distributed to participating laboratories.

This will continue for subsequent years and participants can request to see the results of the tests by emailing nmbaqc@apemltd.co.uk .

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