Particle Size Analysis Component Annual Report
Scheme Operation 2020/2021 (Year 27)

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PARTICLE SIZE COMPONENT ANNUAL REPORT FROM APEM Ltd

SCHEME OPERATION – 2020/21 (Year 27)

Linked Documents (hyperlinked in this report):

Particle Size Exercise Results – PS76

Particle Size Exercise Results – PS77

Particle Size Exercise Results – PS78

Particle Size Exercise Results –PS79

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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 six 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 2019/20; 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 2020/21 PS module exercises (PS76, PS77, PS78 and PS79); seven were government laboratories and ten were private consultancies. Ten laboratories signed up to participate in the PS-OS module exercises (PS-OS19, PS-OS20 and PS-OS21); seven were government laboratories and three were private consultancies. One government laboratory had four Lab Codes to submit twelve 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 2020/21 (Scheme Year 27) 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 cmpleted 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 2020/21 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

Spread-sheet 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 September 2020 each participant was given a confidential, randomly assigned 2020/21 (Scheme year twenty-seven) 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-seven (2020/21) was recorded as PSA_2712.

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, one mud (PS76), two mixed (PS77 and PS78) and one gravel (PS79) were distributed in 2020/21. The samples were distributed in two stages; the first circulation (PS76 and PS77) was sent to participants on 29th October 2020 and the second circulation (PS77 and PS78) was sent on the 10st December 2020. For each circulation participants were given approximately 6 weeks to complete their analysis and send completed workbooks via email to APEM Ltd. PS76 was derived from natural marine sediments; PS78 replicates were prepared from a combination of natural sediments and artificially prepared commercial aggregate and PS77 and PS79 samples were created using artificially prepared commercial aggregate; 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.

3.1.2 Preparation of the Samples

The first PS circulation, PS76, was a mud collected from natural marine environments near the Swanscombe, Kent in The Thames. Approximately 10 litres of visually similar sediment was collected and returned to the laboratory where it was wet sieved at 0.5mm to remove any particles larger than 0.5mm. Sediment that passed through the 0.5mm 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, PS77, was a mixed sample made from comercially aquired aggregate; known amounts of commercially acquired pea shingle (split into half-phi intervals by dry sieving using a mechanical sieve shaker) was mixed with aquired play sand pre-sieved through a 1mm sieve.

The third exercise, PS78, was created from known amounts of commercially acquired Cotswold stone (split into half-phi intervals by dry sieving using a mechanical sieve shaker), commercially acquired play sand pre-sieved through a 1mm sieve to remove any larger particles that may have been present and pre-sieved mud (<0.5mm) mud from The Swale, Yorkshire. The final exercise sample (PS79) was a gravel sample created from known amounts of commercially acquired pea shingle (split into half-phi intervals by dry sieving using a mechanical sieve shaker).

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, 2016)), 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 2020/21 workbooks had the same format as the previous year. Data provided in the "Participant Sieve Metadata" and "Participant Laser Metadata" spreadsheet tabs 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 eight weeks were allowed for the analysis of the first pair of PS samples sent out (PS76 & PS77) and approximately ten weeks for the second pair (PS78 & PS79).

3.2 Results

3.2.1 General comments

Seventeen laboratories subscribed to the exercises in 2020/21. For the first circulation (PS76 and PS77) sixteen subscribing participants provided results; PSA_2710 communicated that they were not participating due to Covid-19 restrictions. PSA_2716 did participate but provided data after the interim reports were issued, their data were incorporated into the final report. For the second circulation (PS78 and PS79) fifteen participants provided results.

PSA_2710 communicated that they were not participating due to Covid-19 restrictions and PSA_2703 communicated that they were not participating as both samples contained a large proportion of gravel and they do not undertake sieve analysis. PSA_2718 appear in PS78, this was a lab participating in a trial to understand whether the scheme was suitable for them and if they would participate in future scheme years.

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 (PS76, PS77, PS78 and PS79) 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 the majority of laboratories are now conducting analyses by laser diffraction the testing of replicates for 2020/21 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 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 PS76 indicated an average composition of 7.93% sand and 92.07% mud, classified as 'Mud' 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. The Benchmark lab used a few drops of Calgon to disperse the sample, as no participant used a chemical dispersant, KPAL re-analysed one of the replictes without dispersant for a more accurate comparison of results.These results can be found in Appendix 5. 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 PS76 Report.

Sample PS77 was a mixed sediment and contained an average of 63.98% gravel, 33.52% sand and 2.50% mud, classified as a 'Gravelly Sand' 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 PS77 Report.

Sample PS78 was also a mixed sediment and contained an average of 56.93% gravel, 29.35% sand and 13.72% mud, classified as a 'Muddy 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 very low variation, with %CV well below the acceptable levels for all 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 PS78 Report.

Sample PS79 was a gravel sample containing an average of 99.98% gravel, 0.01% sand and 0.01% mud. The replicates were analysed by dry sieving and laser analysis although the AQC laboratory observed that usually in an analysis of this kind the small amount of <1mm sediment can be ignored, and the weights above 1mm rescaled to total 100%. However, for completeness, and to ensure the data added up to 100%, the tiny amount of <1mm material was analysed by laser diffraction. The sediment was classified as 'Gravel' according to the Blott & Pye (2012) scheme. There was insufficient material for repeat laser analyses so a single laser subsample was run. The laser triplicate analysis for the single subsample showed low variation, with %CV below the acceptable levels for all 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 PS79 Report.

3.2.3 Results from participating laboratories

In each of the PS76, PS77, PS78 and PS79 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 4. 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 3.

3.2.3.1 Seventy-sixth distribution – PS76

There was generally good agreement for PS76 between the results for the Benchmark replicates and those supplied by the participating laboratories, (see Figure 1). Three participants (PSA_2702, PSA_2913 and PSA_2714) chose to re-submit data after the issue of the interim report. PSA_2702 and PSA_2713 had improved results after re-submission that bought them more in-line with other participant data and the Benchmark data. Although PSA_2714's result improved after re-submission, they still recorded the highest percentage of sand (17.56%). Seven participants recorded the sample as having a Gradistat textural group of 'Mud' and nine participants recorded the sample as 'Sandy Mud'.

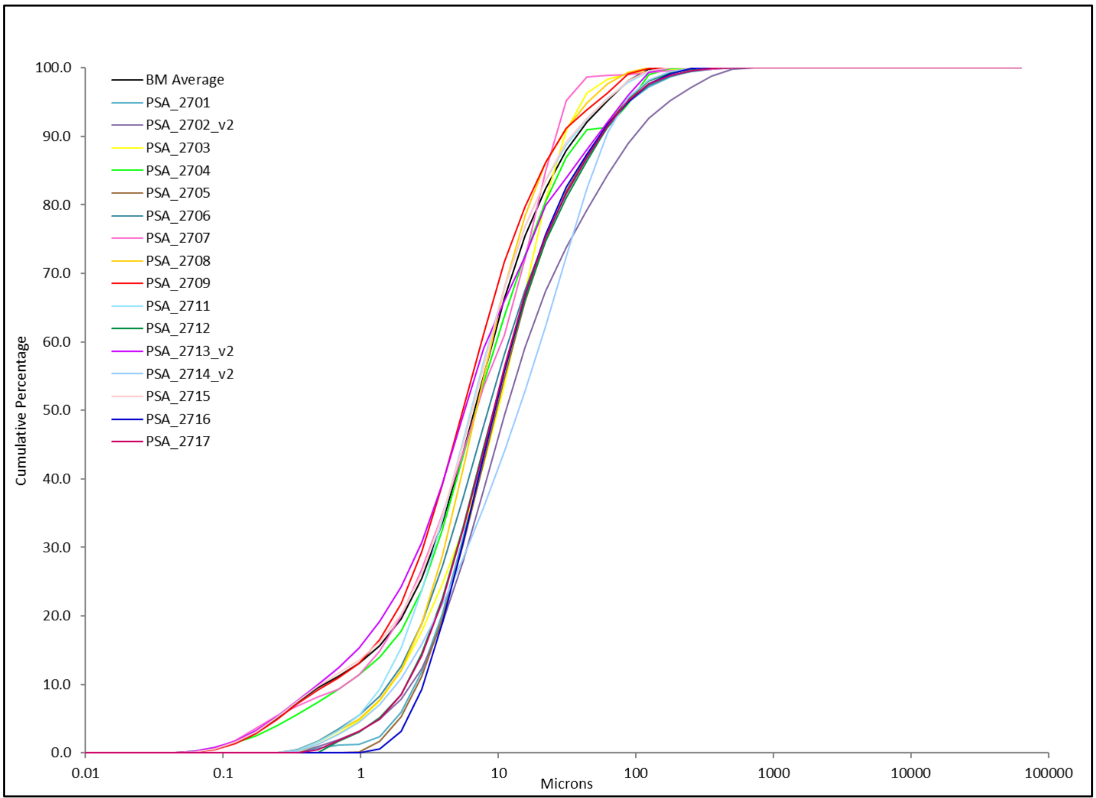

Figure 1. Particle size distribution curves for sediment distributed as PS76 (Figure 6 in PS76).

As recorded in Table 6 of the PS76 report, all laboratories followed the NMBAQC methodology except two participants (PSA_2703 and PSA_2708). All participants analysed the sample using a laser analyser. Table 7 shows that two participants (PSA_2706 and PSA_2713) also undertook sieve analysis; PSA_2706 recorded 0.007g greater than 1mm and PSA_2713 recorded 0.01g in the base pan.

Table 6 also shows the variation in data received from the participating laboratories; the percentage of sand ranged from 1.28% (PSA_2707) to 17.60% (PSA_2714) and percentage mud ranged from 82.40% (PSA_2714) to 98.72% (PSA_2707). No participants used peroxide pre-treatments and one lab (PSA_2715) used a chemical dispersant.

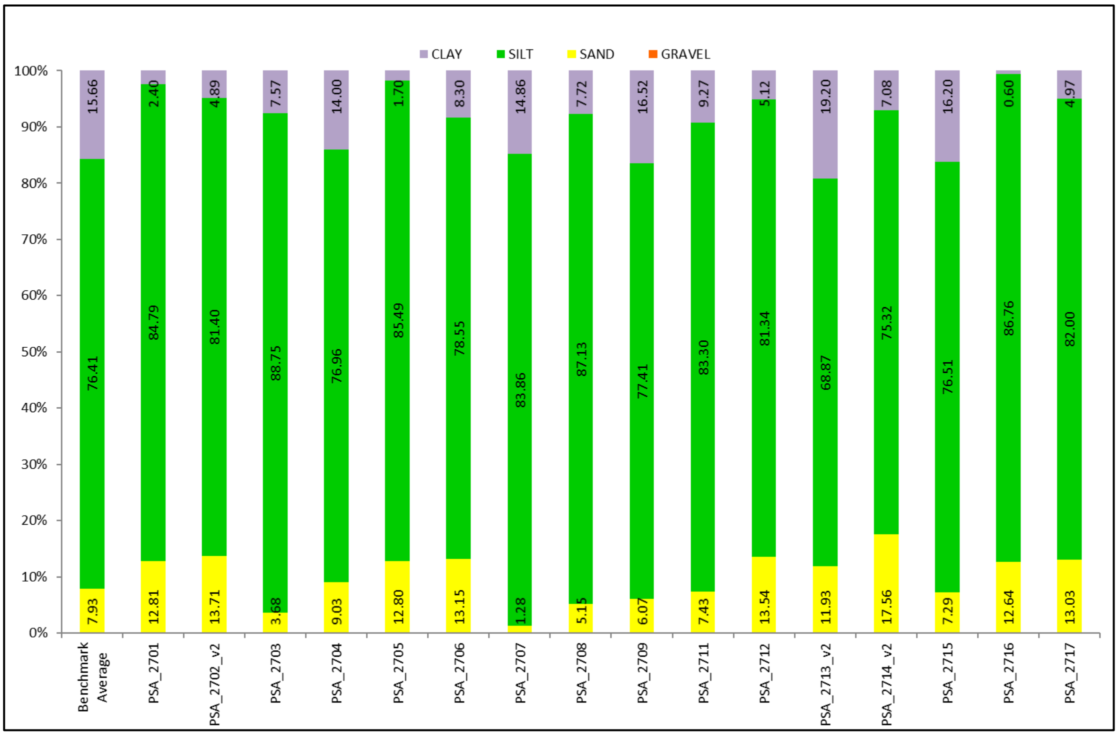

Figure 2. Bar charts showing the percentage gravel, sand, silt and clay for sediment distributed as PS76 (Figure 7 in PS76).

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_2704, PSA_2709, PSA_2713 and PSA_2715 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_2707 is the only laboratory to use a Fritsch laser analyser, which recorded an amount of clay consistent with laboratories using the Beckman Coulter instruments. Of those participants using Malvern instruments, only two (PSA_2708 and PSA_2711) recorded a similar proportion of Sand to the Benchmark Data; 5.15% and 7.43%, respectively whilst the Benchmark Data recorded an average of 7.93%.

3.2.3.2 Seventy-seventh distribution – PS77

There was good agreement for PS77 between the results from the analysis of the benchmark replicates and those from the participating laboratories (see Figure 3). Participant PSA_2705 re-submitted their data after the interim report had been issed as they had enetered their laser data inverted. The majority of the participants had a Gradistat textural group of ‘Sandy

Gravel'. The exceptions were PSA_2706, PSA_2707, PSA_2714 and PSA_2716 whose results were classified as 'Muddy Sandy Gravel'. Participant PSA_2703 does not subscribe to the sieve aspect of the module and therefore are assed on the laser analysis only. The percentage of gravel recorded by the participants (see Figure 4), ranged from 60.50% (PSA_2707) to 65.34% (PSA_2701). The percentage of sand ranged from 29.20% (PSA_2706) to 35.45% (PSA_2707) and the percentage mud ranged from 1.46% (PSA_2712) to 6.89% (PSA_2706).

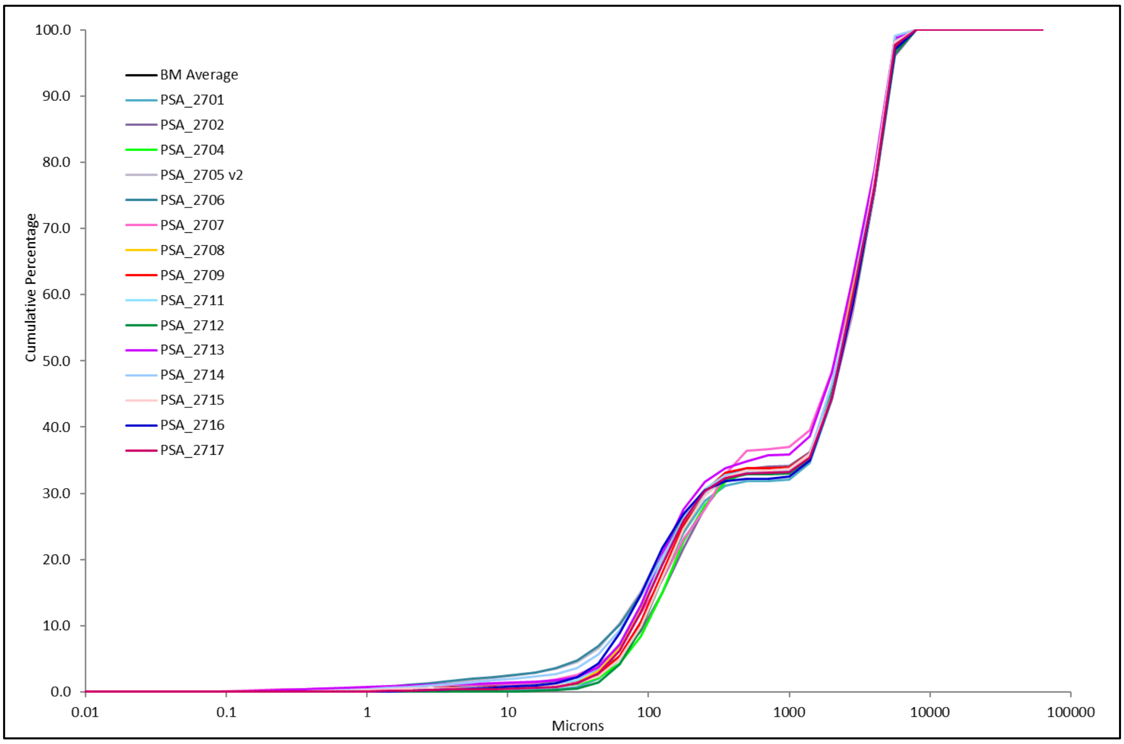

Figure 3. Particle size distribution curves for sediment distributed as PS77.

Appendix 2 shows that laser manufacturer did not have much of an impact on the results; all participants recorded less than 0.8% clay with only participants PSA_2701 and PSA_2712 recording no clay content at all.

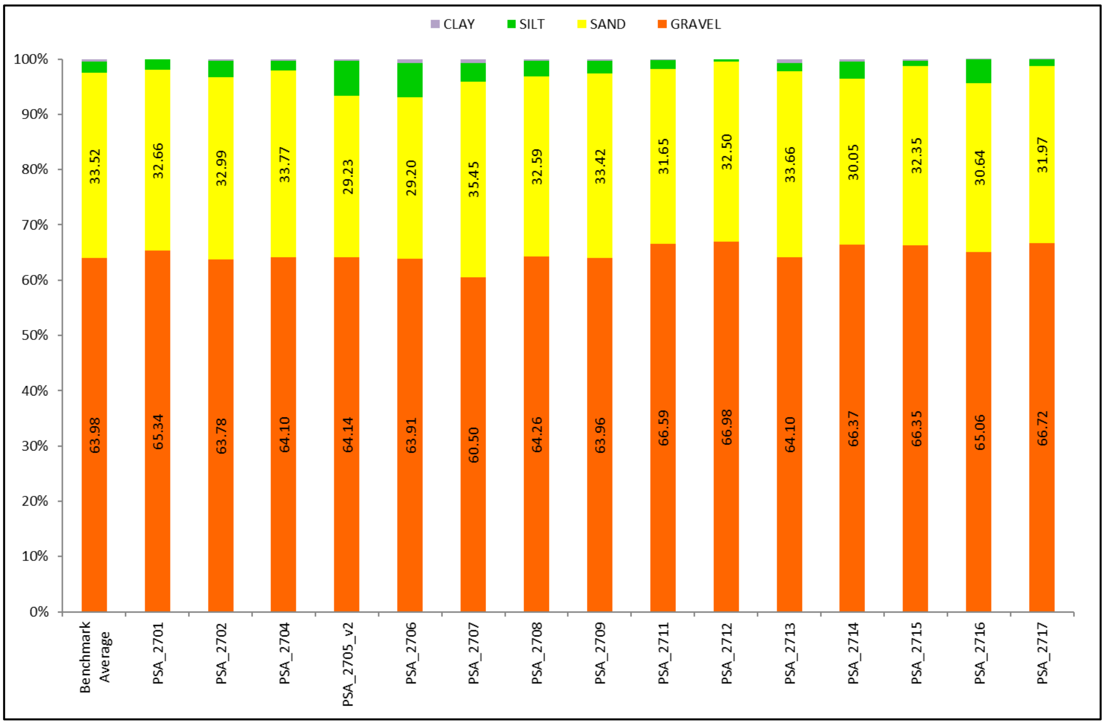

Figure 4. Bar charts showing the percentage gravel, sand, silt and clay for sediment distributed as PS77 (Figure 7 in PS77).

3.2.3.3 Seventy-eighth distribution – PS78

There was generally good agreement for PS78 between the results reported by the participating laboratories and those obtained for the benchmark replicates, as seen in Figure 5 and 6. Participant PSA_2718 is a perspective new participant undertaking a trial;due to this their results will not be included in any details provided in this report. Table 6 in the PS78 report shows that all participants used both sieve and lase analysis and no participants used chemical dispersants or peroxide pre-trratments. All participants recorded a Gradistat textural group of 'Muddy Sandy Gravel'. Percentage gravel ranged from 51.70% (PSA_2707) to 59.95% (PSA_2705), the percentage of sand ranged from 28.40% (PSA_2705) to 35.72% (PSA_2709) and the percentage of mud ranged from 8.06% (PSA_2709) to 15.30% (PSA_2704).

The result for PSA_2707 has recorded a slightly lower percentage of gravel (51.7%) compared to the other participants (average of 57.3%), Figure 7 in the P78 report also shows that they recorded the highest weight of sediment <1mm (320.11g), although this had little effect on the overall description of the sample which is described as Muddy Sandy Gravel as per the other results.

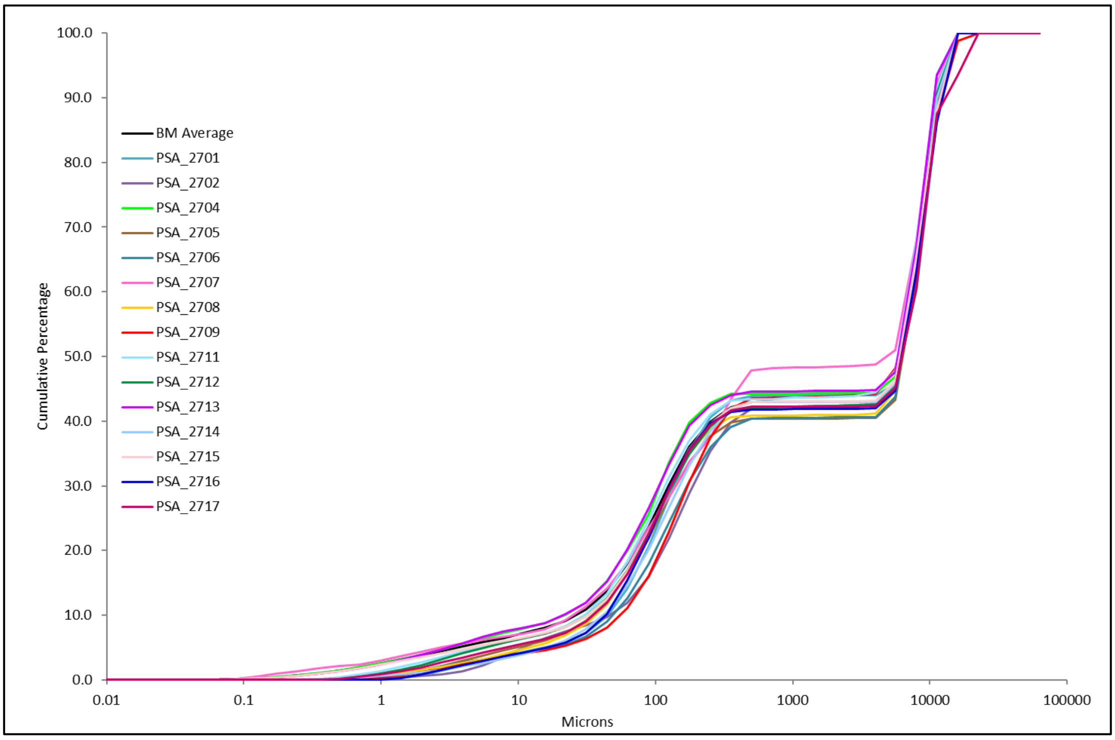

Figure 5. Particle size distribution curves for sediment distributed as PS78 (Figure 6 in PS78).

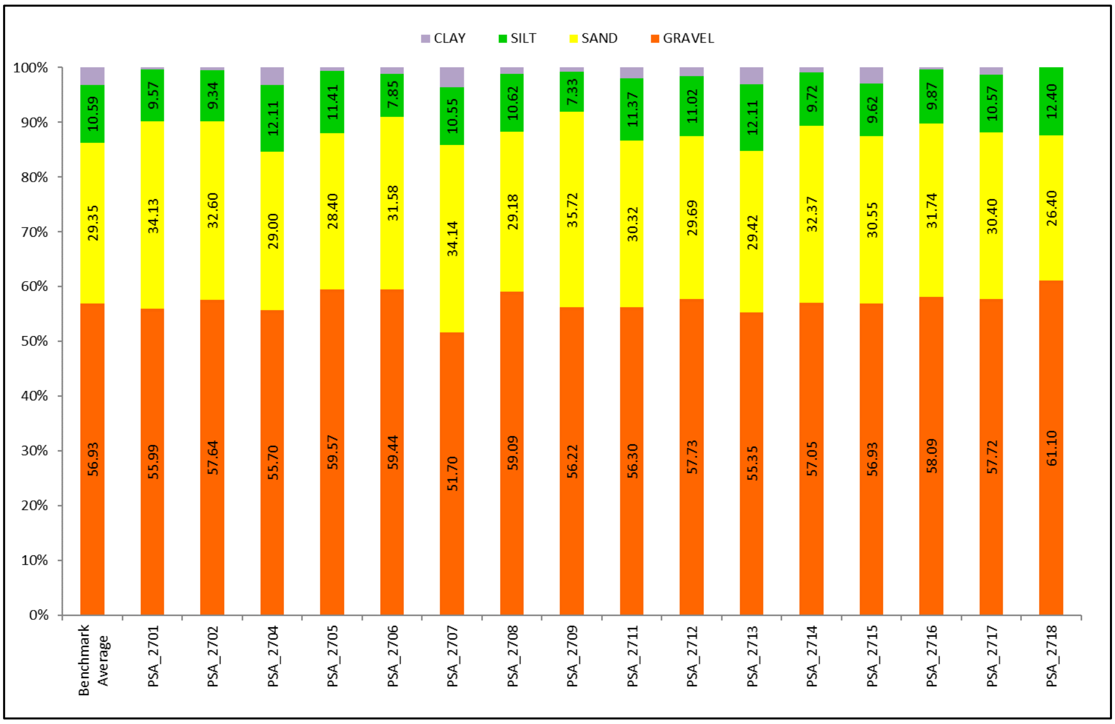

Figure 6. Bar charts showing the percentage gravel, sand, silt and clay for sediment distributed as PS78 (Figure 7 in PS78).

3.2.3.4 Seventy-ninth distribution – P79

There was very good agreement in results between the laboratories and the benchmark data (see Figure 7). All participants had a Gradistat textural group of 'Gravel', with an average of 99.93% Gravel. The sample was supplied as a dry sample; this may have caused some confusion as it would not be possible to undertake a wet separation at 1mm as stated by the NMBAQC methodology. As a result of this the sample only required dry sieve analysis.

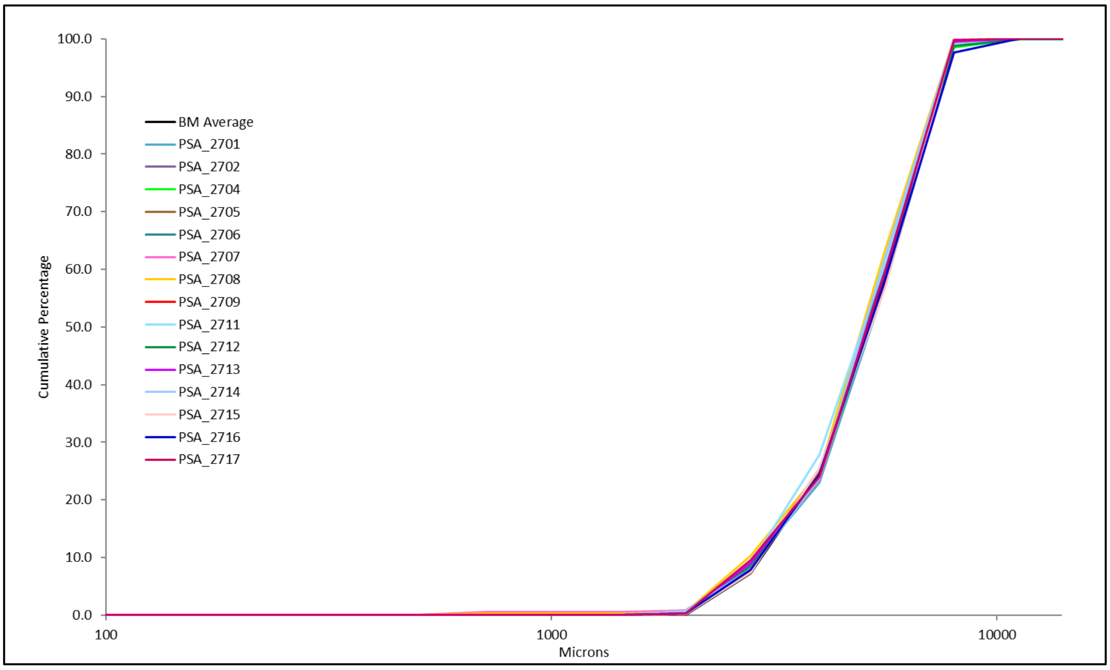

Figure 7. Particle size distribution curves for sediment distributed as PS79 (Figure 7 in PS79).

For those participants following the NMBAQC methodology and dry sieving to 1mm the process produced some less than 1mm material that was collected in the base pan. This base pan weight can either be incorporated into the final data in the 0.0 to 0.5 phi size interval or excluded and data rescaled to 100%. Either of these approaches is acceptable. Participant PSA_2712 was the only participant who excluded the base pan weight but did not re-scale the data meaning that their final data set did not sum to 100%.

The benchmark laboratory commented: "usually in an analysis of this kind [the small amount of <1mm retained in the base pan] can be ignored, and the weights above 1 mm rescaled to total 100%. However, for completeness, and to ensure this spreadsheet totals 100%, this tiny amount of material was analysed by laser diffraction and entered into the laser sheet." The Benchmark data recorded an average of 0.01% Mud. Two participants (PSA_2706 and PSA_2715) chose to carry out laser analysis on the less than 1mm base pan fraction, recording between 0.01% (PSA_2715) and 0.04% (PSA_2706) Mud. Although laser processing was not required, undertaking it had little effect on the overall sample profile.

3.2.4 Discussion

The exercise reports show that the majority of participants follow the NMBAQC methodology for these exercises; those that do not, do so for genuine reasons. PSA_2703 do not undertake analysis of sediment greater than 1mm so chose to only participate in the laser analysis for PS76 and PS77 and opted out of PS78 and PS79.

3.2.4.1 Sieve Analysis (>1mm)

The three exercises that contained larger quantities of sediment greater than 1mm (PS77, PS78 and PS79) show that the dry sieve analysis (>1mm) undertaken by participants was generally in agreement with each other and the benchmark data (see Figure 8). 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 and amplitude of 1.5nm/'g'. Of the sieve metadata provided by participants, sieve time varies from 5 to 30 minutes, sieve amplitude is not provided by many participants and often doesn'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.

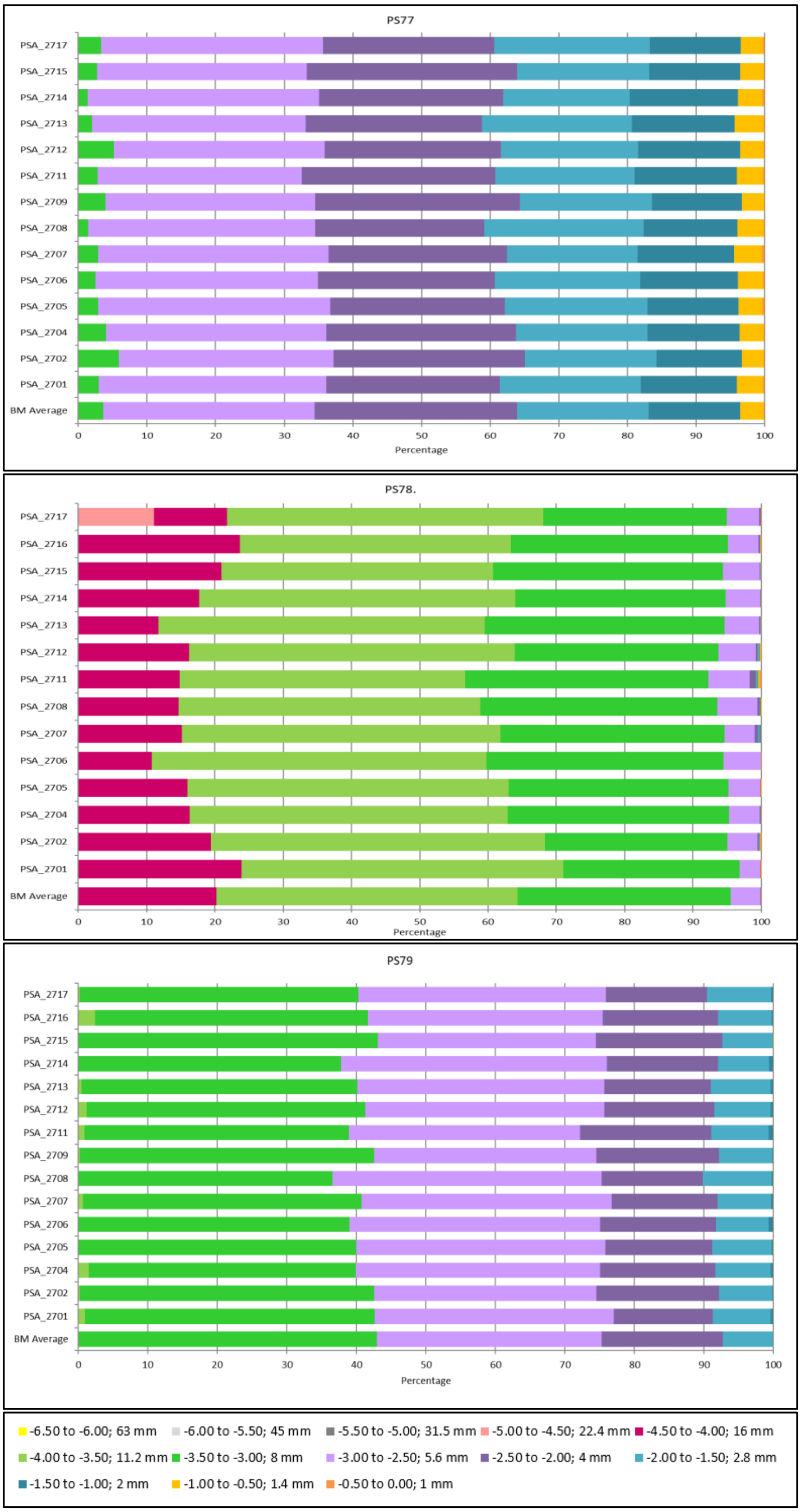

Figure 8. Bar charts showing raw sieve data as percentage in each half-phi interval for PS77, PS78 and PS79.

3.2.4.2 Laser Analysis (<1mm)

Figure 9 shows the cumulative and differential curves for the laser data for each exercise that contained a significant amount of material less than 1mm. Although the results continue to show improvement from previous years, laser analysis remains the main source of variability between participants. The majority of participants now remember to re-scale laser data to 100%; Table 8 in each of the exercise reports shows if the final laser data has been re-scaled or not. Generally, where data has not summed to 100% it appears to be due to data entry or rounding errors. For PS76 and PS78 all participants provided final laser data that had been re-scaled to 100%. In exercise PS77, final laser data for participant PSA_2705 and PSA_2707 both sum to 99.98%, however both have been re-scaled to produce the final merged data. Laser analysis was not required for exercise PS79 due to the small amount of <1mm material present, but all participants who undertook laser analysis correctly rescaled their data to 100%.

As in previous years it was apparent in the exercises that required laser analysis and had a significant mud fraction (PS76) that there were 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. There were still slight differences detected between the participants using Coulter instruments, however, and these could be due to differences in the samples supplied to each lab, different sub-sampling, sample dispersion and/or sample presentation procedures being used. The Malvern instruments generally recorded a slightly higher percentage of sand (average of 11.40%) compared to other instruments, average for Beckman instruments was 8.45%, the Fritsch instrument only recorded 1.28% sand.

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.

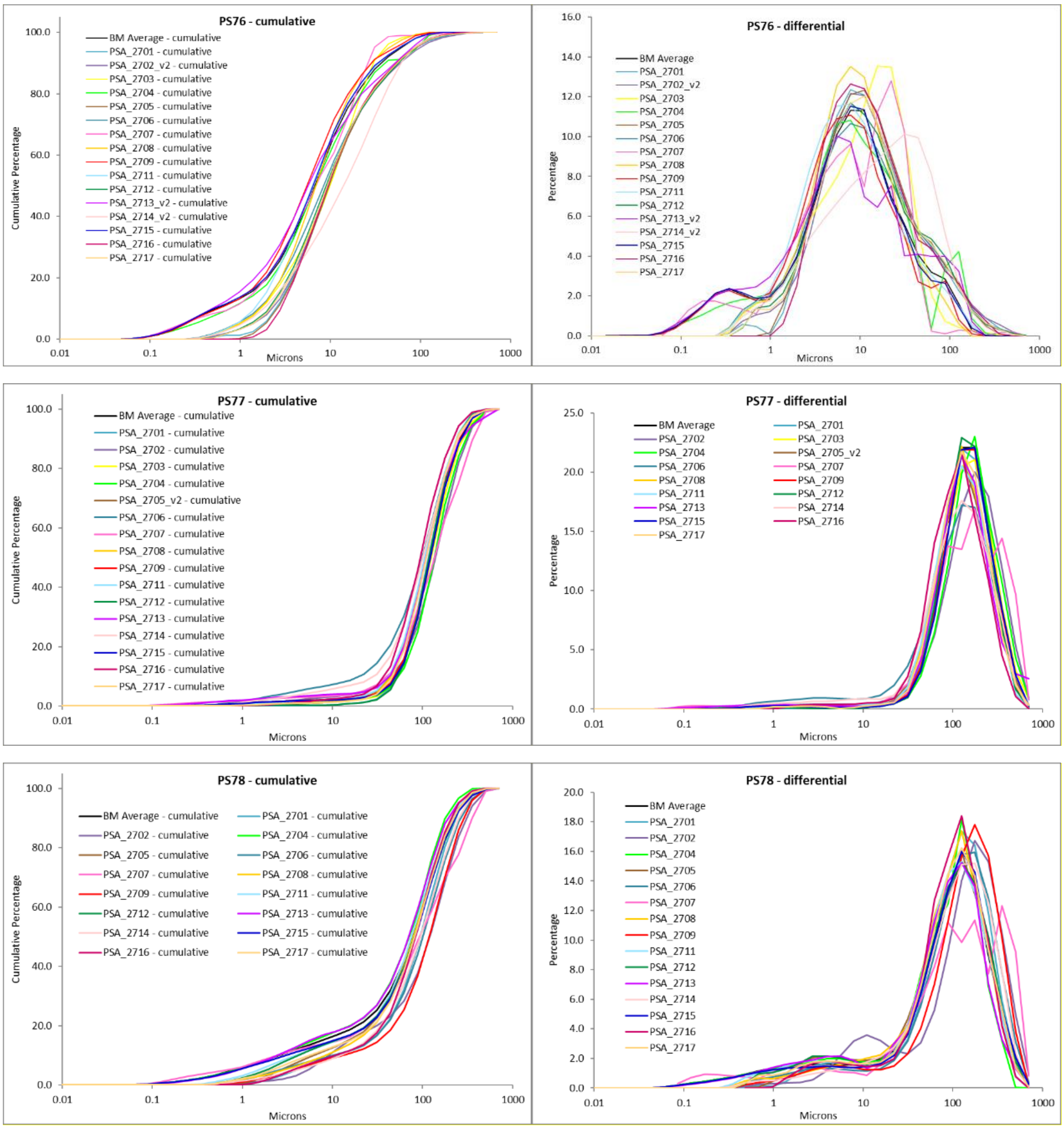

Figure 9. Cumulative and differential final laser data provided by participants for exercises PS76, PS77 and PS78.

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. This year's workbooks used the same format as last year; the metadata section had been made simpler for participants as they just had to complete a form from a set of drop-down menus. Thus, the majority of participants supplied laser metadata in the current year, PSA_2708 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.

For Exercise PS76, PS77 and PS78 all the participants that submitted metadata are now using the Mie Theory analysis model. All but two of the participants that provided metadata information used a Particle Absorption Index of 0.1, the two exceptions were PSA_2701 and PSA_2705, who used a Particle Absorption Index of 0.01. Most participants used a Particle Refractive Index of 1.55, although there were variations; 1.52 (PSA_2701, PSA_2703 and PSA_2714), 1.56 (PSA_2705) and 1.59 (PSA_2716). 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 except for PSA_2705 who used the red light only.

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 2012/21 (Scheme year 27) 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 issed 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 it's seventh 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.

Spread-sheet 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). 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.

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.

Due to the Covid-19 pandemic the timetable for the PS-OS module was altered giving participants more time to submit data. Participants who took part in Batch 1 submited data by 13th November 2020, had samples selected by the 11th December 2020 and reports were issued by the end of March 2021, Batch 2 submited data by 31st March 2021, had samples selected by the 16th April 2021 and reports were issued by the end of June 2021

4.2 Results

4.2.1 General comments

Eleven laboratories subscribed to the PS-OS module in 2020/21. One of the eleven labs had four lab-codes to facilitate multiple PS-OS submissions. Ten of the eleven laboratories that subscribed to the module provided data and nine submitted samples for re-analysis. Participant_2710 chose not to participate due to Covid-19 restrictions and Participant PSA_2709 submitted data from which samples were slected however despite several reminders no samples or completed data sheets have been received and as such they could not be included in this report.

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; exceptions to this were participants PSA_2714 and PSA_2721. PSA_2714 did not provide any laser sub-samples, therefore after weighing, the dried <1mm fractions were used for laser analysis. This required re-wetting and mixing into a soft but stiff paste consistency in order to extract representative laser subsamples. PSA_2721 appeared to have only provided the bulk sample, with no <1 mm and >1mm components, therefore a standard NMBAQC methodology was undertaken.

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

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_2711	PS-OS 19	Gravelly Mud	Gravelly Mud
	PS-OS 20	Gravelly Muddy Sand	Gravelly Muddy Sand
	PS-OS 21	Slightly Gravelly Sandy Mud	Slightly Gravelly Sandy Mud
PSA_2712	PS-OS 19	Slightly Gravelly Sand	Slightly Gravelly Sand
	PS-OS 20	Slightly Gravelly Sand	Slightly Gravelly Sand
	PS-OS 21	Slightly Gravelly Sand	Slightly Gravelly Sand
PSA_2713	PS-OS 19	Slightly Gravelly Sand	Slightly Gravelly Sand
	PS-OS 20	Gravel	Gravel
	PS-OS 21	Gravelly Muddy Sand	Gravelly Muddy Sand
PSA_2714	PS-OS 19	Gravelly Muddy Sand	Gravelly Muddy Sand
	PS-OS 20	Gravelly Sand	Gravelly Muddy Sand
	PS-OS 21	Gravelly Mud	Gravelly Mud
PSA_2715	PS-OS 19	Slightly Gravelly Sand	Slightly Gravelly Sand
	PS-OS 20	Slightly Gravelly Sand	Slightly Gravelly Sand
	PS-OS 21	Gravelly Sand	Gravelly Sand
PSA_2717	PS-OS 19	Slightly Gravelly Muddy Sand	Slightly Gravelly Muddy Sand
	PS-OS 20	Slightly Gravelly Sandy Mud	Slightly Gravelly Sandy Mud
	PS-OS 21	Slightly Gravelly Sand	Slightly Gravelly Sand
PSA_2718	PS-OS 19	Slightly Gravelly Sand	Slightly Gravelly Muddy Sand
	PS-OS 20	Slightly Gravelly Muddy Sand	Slightly Gravelly Sandy Mud
	PS-OS 21	Slightly Gravelly Sandy Mud	Slightly Gravelly Sandy Mud
PSA_2719	PS-OS 19	Slightly Gravelly Sand	Slightly Gravelly San
	PS-OS 20	Gravelly Mud	Muddy Gravel
	PS-OS 21	Muddy Gravel	Muddy Gravel
PSA_2720	PS-OS 19	Muddy Sandy Gravel	Muddy Sandy Gravel
	PS-OS 20	Sandy Gravel	Sandy Gravel
	PS-OS 21	Sandy Gravel	Muddy Sandy Gravel
PSA_2721	PS-OS 19	Sand	Slightly Gravelly Sand
	PS-OS 20	Muddy Sand	Slightly Gravelly Muddy Sand
	PS-OS 21	Sand	Slightly Gravelly Sand
PSA_2722	PS-OS 19	Slightly Gravelly Sand	Slightly Gravelly Muddy Sand
	PS-OS 20	Slightly Gravelly Muddy Sand	Slightly Gravelly Muddy Sand
	PS-OS 21	Gravelly Sand	Gravelly Sand

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.

Participant PSA_2721 provided data that did not contain any sediment greater than 1mm however during the AQC analysis some >1 mm particles and shells were retained on the 1mm mesh. Therefore, a standard NMBAQC analysis was performed on these samples. Several large shells and shell fragments were found. Thus, the AQC results included the >1 mm component causing all samples to be classified as "Sllightly Gravelly".

Sample PS-OS 20 from participant PSA_2719 had an issue where the oven dried less than 1mm weights recorded by the primary analysis and the AQC differed by 50 grams causing the AQC data to have an elevated Gravel content. At the time of writing this report the AQC lab had doubled checked the weight they had recorded however no explaination or correction has been provided by the primary laboratory.

Small amounts of variability particularly in percentage clay shown in Figure 10 and 11 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. Participants PSA_2711, PSA_2712, PSA_2715, PSA_2717, PSA_2719 and PSA_2721 display these slight differences however they are not significiant enough to alter the sample textural group seen in Table 1.

Participant PSA_2714 displays differences from the AQC analysis in sand, silt and clay content in all three samples (see Table 2). The differences in PS-OS 20 cause the original analysis and the AQC analysis to have differing sediment textural groups (see Table 1); the participant recorded the sample as Gravelly Sand and the AQC as Gravelly Muddy Sand.

	Primary PS-OS 19	AQC PS-OS 19	Primary PS-OS 20	AQC PS-OS 20	Primary PS-OS 21	AQC PS-OS 21
Gravel	22.38	22.46	15.57	15.21	17.21	16.96
Sand	57.71	41.61	77.36	63.74	26.50	16.53
Silt	18.64	29.87	7.07	18.95	52.34	54.87
Clay	1.26	6.06	0.00	2.11	3.95	11.64

Table 2. Percentage Gravel, Sand, Silt and Clay recorded in PS-OS 19, 20 and 21 for participant PSA_2714 and the AQC lab. Data used to create bar charts seen in Figure 10.

Table 2 shows that for PS-OS 20 the AQC analysis recorded 13.62% less sand, 11.88% more silt and 2.11% more clay than the original analysis undertaken by PSA_2714. These differences are considered too big to be caused solely by differing laser analysis and could be caused by poor sample preparation, poor homogenisation or presentation to the laser. The AQC lab also commented that the laser subsample was clearly not at field state and had not been taken from the bulk sediment. It was composed of oven dried sediment and contained small dried aggregates of mud. It is likely this was a small part of the <1mm oven dried fraction re-labelled as a laser subsample. The material required wetting, soaking, and ultrasonic treatment to adequately disperse the mud aggregates before analysis. If the original laser analysis was performed on a subsample of the bulk sediment before drying then it would be expected that this re-analysis would be different, as subsequent sieving and oven drying will inevitably change the particle size.

For PSA_2718 (PS-OS 19 and 20) and PSA_2720 (PS-OS 21) the primary and AQC analysis have different textural groups (see Table 1). However the differences in sand and mud content are approximately only 5% and it is just that the differences occur around the boundary limits of worded textural descriptions.

The textural group difference seen in PSA_2722 PS-OS 19 are most likely due to differing methodologies between the participant and the Benchmark Lab; the participant does not have a laser analyser so cannot analyse the samples following the NMBAQC methodology.

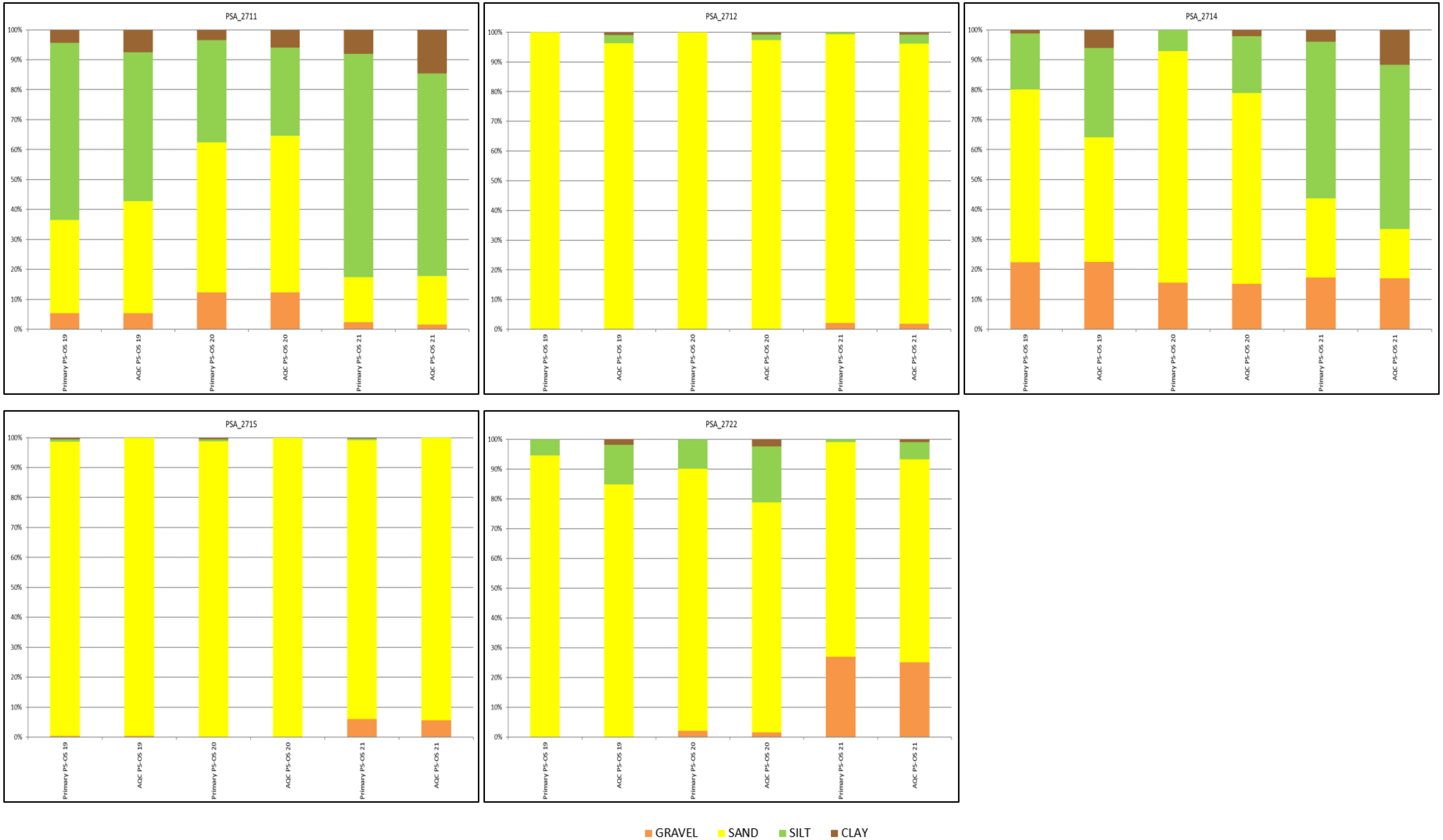

Figure 10. Bar charts showing percentage gravel, sand, silt and clay from laboratories participating in the PS-OS module (Batch 1).

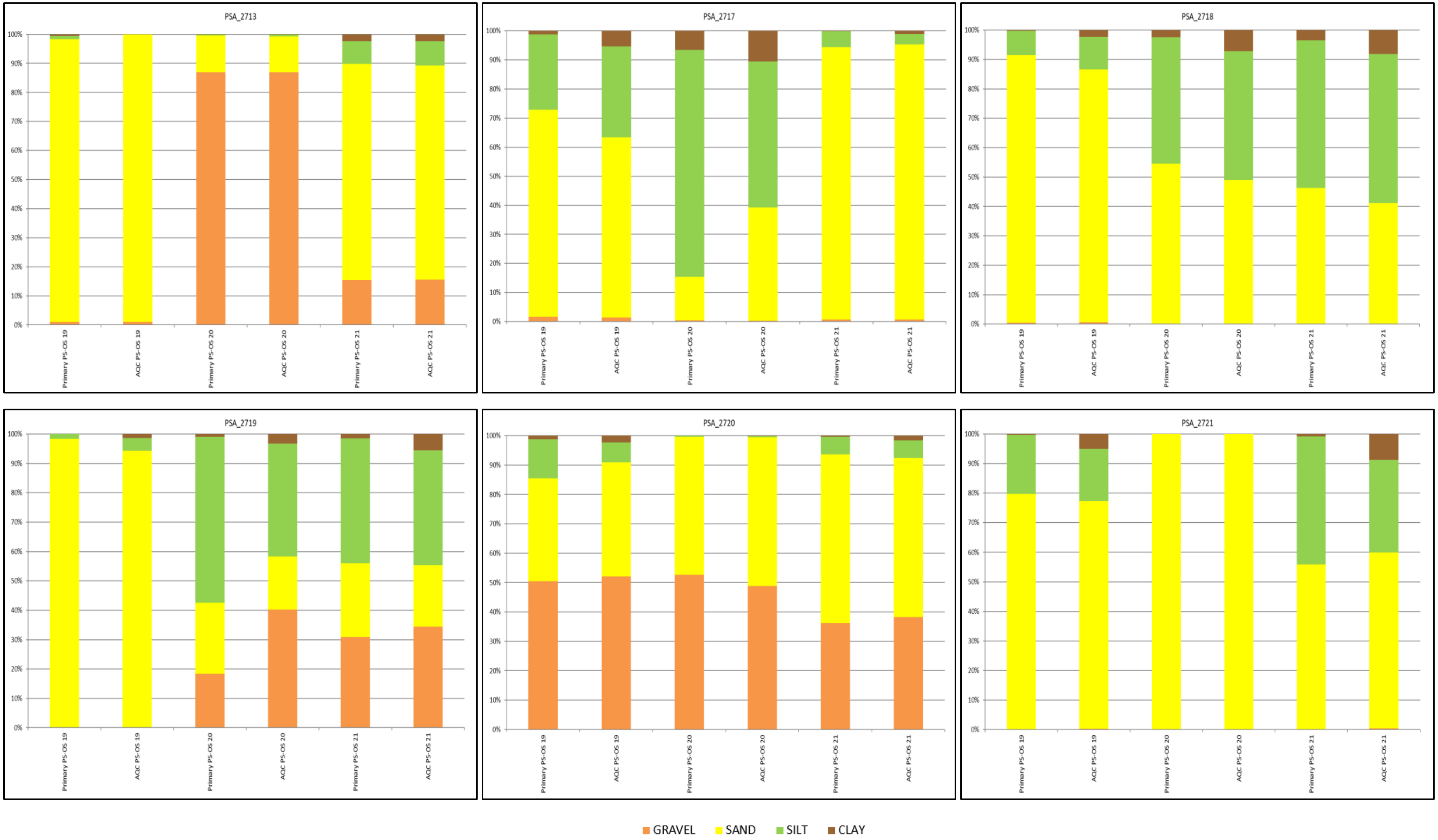

Figure 11. . Bar charts showing percentage gravel, sand, silt and clay from laboratories participating in the PS-OS module from Batch 2.

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, 2016), 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 the vast majority of 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 (2016).

The returns for the 2020/21 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 that all participants are getting comparable results when they follow the same methodology. It is therefore important that only the NMBAQC methodology (Mason, 2016) 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 are all filled in correctly. This will help eradicate typing and transcription errors. The workbook has been updated for the next Scheme Year (Year 28) to help enable the continuity of data through the workbook. Conditional formatting will flag 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. Lydia McIntyre-Brown (APEM), Scheme contract manager Claire

Mason (Cefas) and Jon Barry (Cefas) are currently researching a statistical method to compare participant results with the Benchmark data. Although this year's data is not ready to be trialled yet there is the possibility of a report detailing the outcomes available in the next couple of scheme years.

4. **<u>Possible workshop looking at sample preparation and presentation to laser.</u>** Covid-19 restrictions put an end to any possible face to face workshops in Year 27, as restrictions ease this may become an option in the next couple of Scheme Years.

   Most participants now use the recommended laser parameters of an optical model of Mie Theory with Particle Refractive index of 1.55 and a Particle Absorption Index of 0.1; however, the results can still differ from the Benchmark data and other participants. One possible reason for this could be due to sample preparation and homogenisation as well as presentation of the sample to the laser. Another issue that has occurred is whether muddy samples need only laser analysis or whether sieve analysis should be undertaken too. There were incidents where participants recorded less than 1g of sediment greater than 1mm causing sample descriptions to become "slightly gravelly". 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**.** A workshop, either in person or a webinar detailing how to create and homogenise a laser sub-sample, particularly looking at the use of ultrasonics may be useful in forth coming years.

5. **<u>Health and Safety.</u>** 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. 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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