Particle Size Analysis Component Annual Report
Scheme Operation 2018/2019 (Year 25)

Authors:	Lydia McIntyre-Brown (APEM), NMBAQCS Particle Size Analysis Administrator
	Prof. Kenneth Pye (KPAL), NMBAQCS Particle Size Benchmark Analyst
Reviewer:	David Hall (APEM), NMBAQCS Project Manager
Approved by:	Claire Mason (Cefas), Contract Manager
Contact:	nmbaqc@apemltd.co.uk

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PARTICLE SIZE COMPONENT ANNUAL REPORT FROM APEM Ltd

SCHEME OPERATION – 2018/19 (Year 25)

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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 comprises of 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 component of the scheme comprises of two modules:

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

The PS module followed the same format of 2017/18; 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.

Sixteen laboratories signed up to participate in the 2018/19 PS module exercises (PS68, PS69, PS70 and PS71); six were government laboratories and ten were private consultancies. Ten laboratories signed up to participate in the PS-OS module exercises (PS-OS13, PS-OS14 and PS-OS15); five were government laboratories and five were private consultancies. Two government laboratories had two Lab Codes to submit six PS-OS samples each 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 2018/19 (Scheme year 25) both the PS and PS-OS reports will follow a similar format with each sample analysis section broken down for review, including sieve processing, laser processing, data merging and summary statistics. Laboratories will then receive a "Good" or "Review" flag based on their results; "Review" flags will have accompanying comments as to where mistakes have been made and how to correct them.

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. 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 2018/19 year PSA modules, PS and PS-OS are described in more detail below. A brief outline of the information to be obtained from the module is given, together with a description of the preparation of the necessary materials and brief details of the processing instructions given to each of the participating laboratories.

2.2 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 were distributed to each participating laboratory via email for each circulation 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 August 2018 each participant was given a confidential, randomly assigned 2018/19 (Scheme year twenty-five) 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-five (2018/19) was recorded as PSA_2512.

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 (PS68), one sandy (PS70) and two mixed (PS69 and PS71) were distributed in 2018/19. The samples were distributed in two stages; the first circulation (PS68 and PS69) was sent to participants on 31st August 2018 and the second circulation (PS70 and PS71) was sent on the 30th November 2018. For each circulation participants were given approximately 6 weeks to complete their analysis and send completed workbooks via email to APEM Ltd. PS68 was derived from natural marine sediments; PS70 was derived from commercially acquired material and PS69 and PS71 replicates were prepared from a combination of natural sediments and artificially prepared commercial aggregate; they were prepared at APEM's Letchworth laboratory as described below.

3.1.1 Preparation of the Samples

The first PS circulation, PS68, was a mud collected from natural marine environments at Gweek Quay, Helford River. Approximately 20 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, any excess water was removed before it was cored into replicate samples approximately 200 grams in weight. The second exercise, PS69, was a mixed sample created from known amounts of commercially acquired pea shingle (split into half-phi intervals by dry sieving using a mechanical sieve shaker) with known quantities of sand from near the Cutty Sark, Greenwich, Thames Estuary. The sand was pre-sieved through a 1mm sieve to remove any larger particles before being mixed and left to dry out. The third sediment (PS70) was created using commercially acquired builders sand pre-sieved through a 1mm sieve to remove any larger particles that may have been present. The final exercise sample (PS71) was a diamicton sample made from natural sediments consisting of pre-sieved (<1.0mm) sand from Shoreham-on-Sea, East Sussex, and a pre-sieved (<0.5mm) mud from Gweek Quay, Cornwall as well as known weights of commercially acquired pea shingle.

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 produce benchmark data. Where laser diffraction analysis was required, these replicates were analysed using a 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.

3.1.2 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 data set.

The 2018/19 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 six weeks were allowed for the analysis of each pair of PS samples sent out (i.e. PS68 & PS69, PS70 & PS71).

3.2 Results

3.2.1 General comments

Sixteen laboratories subscribed to the exercises in 2018/19. For the first circulation (PS68 and PS69) all subscribing participants eventually provided results; PSA_2515 were given an extension due to a laser malfunction needing an engineer to fix. For the second circulation (PS70 and PS71) all but one participant provided results. PSA_2506 did not participate in exercises PS70 or PS71 and did not provide email confirmation of their non-participation. Participant PSA_2513 was given an extension due to delays with their sub-contractor.

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 (PS68, PS69, PS70 and PS71) 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 2018/19 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 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. Good laser reproducibility was shown for replicates when the %CV was <3% for the D_{50} and <5% for the D_{10} and D_{90}, all limits were doubled when the D_{50} was less than 10µm, in line with recommendations in BS ISO 13320.

Benchmark analysis of the replicates for Sample PS68 indicated an average composition of 20.56% sand and 79.44% mud, classified as “Sandy 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} andD_{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 PS68 Report.

Sample PS69 was a mixed sediment and contained an average of 23.91% gravel, 74.89% sand and 1.21% 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 PS69 Report.

Sample PS70 was a sand sample that required laser analyses only. The sample contained an average of 90.10% sand and 9.90% mud and was classified as ‘Sand’ according to the Blott & Pye (2012) scheme. The laser triplicate analysis generally showed low variation, with %CV below the acceptable levels for most 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 PS70 Report.

Sample PS71 was a mixed sample and required sieve and laser analysis. The results showed an average of 35.19% gravel, 57.32% sand and 7.49% mud. The sediment is classified as ‘Muddy Sandy Gravel’ according to the Blott & Pye (2012) scheme. The laser triplicate analysis 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 PS71 Report.

3.2.3 Results from participating laboratories

In each of the PS68, PS69, PS70 and PS71 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 five 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; in the third table the final laser data submitted by each participant is shown. The fourth and fifth tables show the results of the triplicate laser analysis supplied and the Coefficient of Variance of the D_{10}, D_{50} and D_{90}. 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 displayed in Figure 7. The final merged data submitted by each participant and the benchmark laboratory are provided in Appendix3.

3.2.3.1 Sixty-eighth distribution – PS68

There was generally good agreement for PS68 between the results for the Benchmark replicates and those supplied by most of the participating laboratories, (see Figure 1).

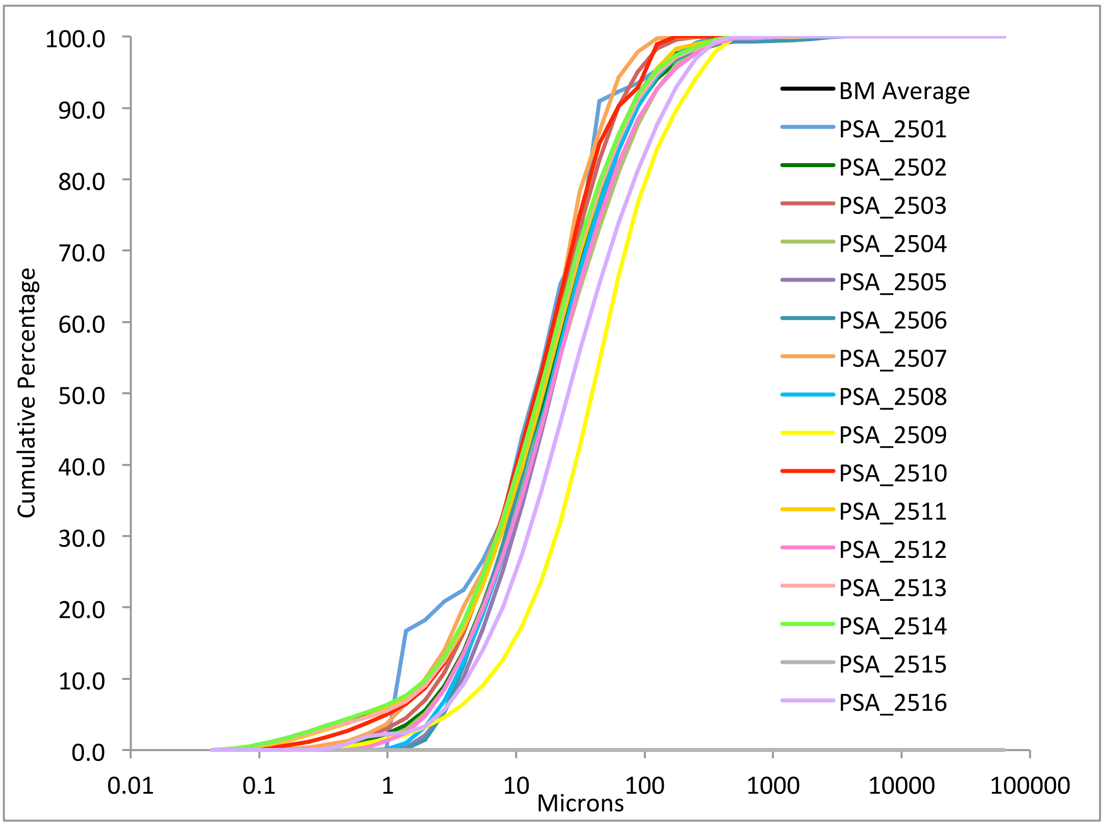

Figure 1. Particle size distribution curves for sediment distributed as PS68 (Figure 6 in PS68).

The result for PSA_2501 follows a slightly different distribution to other participants as they do not have access to a laser analyser and therefore are following a different methodology as stated in Table 6 in the PS68 Report.

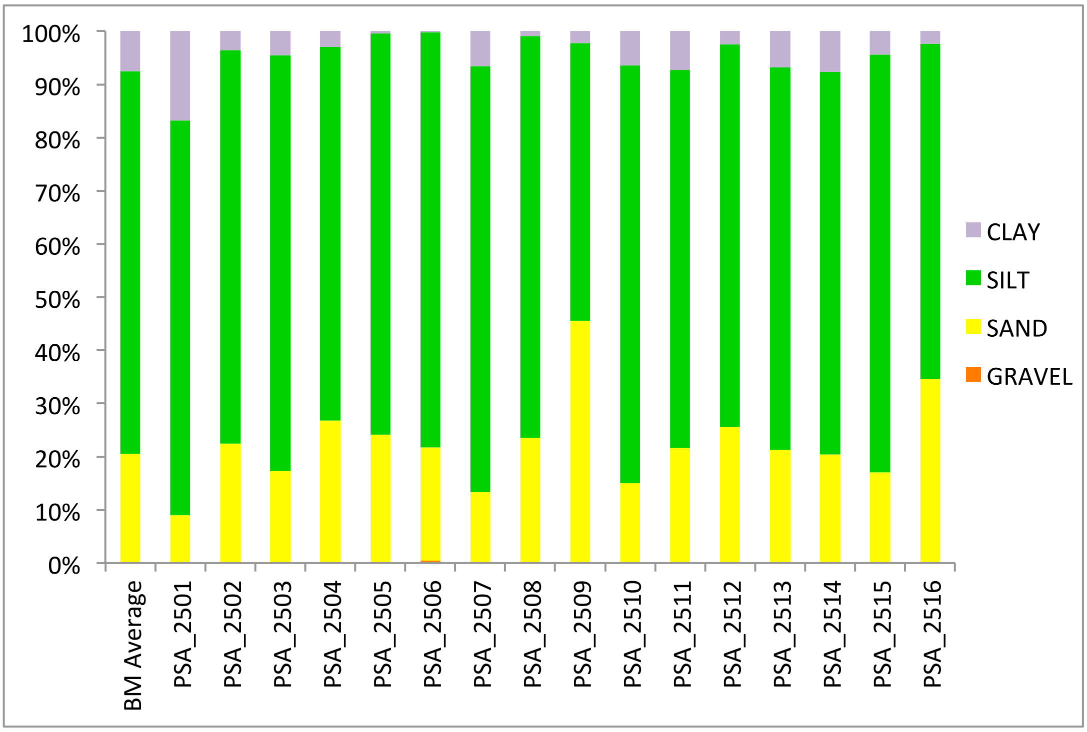

Figure 2. Bar charts showing the percentage gravel, sand, silt and clay for sediment distributed as PS68 (Figure 7 in PS68).

Table 6 also shows the variation in data received from the participating laboratories; of the labs using a laser analyser the percentage of sand ranged from 13.4% (PSA_2507) to 45.9% (PSA_2509) and percentage mud ranged from 54.1% (PSA_2509) to 86.6% (PSA_2507). No participants used peroxide pre-treatments; two participants PSA_2501 and PSA_2502 used chemical dispersants. Of those following the NMBAQC methodology five participants (PSA_2506, PSA_2508, PSA_2511, PSA_2512 and PSA_2516) chose to undertake sieve and laser analysis on this sample, the remainder only undertook laser analysis. Those that undertook sieve analysis found small amounts (0.04g – 0.41g) of sediment greater than 1mm, equating to a gravel percentage of 0.03% to 0.5% of the total sample. 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.

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_2510, PSA_2511, PSA_2513 and PSA_2514 as well as the Benchmark Lab use the Beckman Coulter LS13 320 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).

Of the participants following the NMBAQC methodology, the percentage sand was generally around 20-25% and the percentage silt around 70-75% for the majority of participants; three participants (PSA_2507, PSA_2509 and PSA_2516) recorded percentages of sand and silt that were substantially different from this. Participant PSA_2507 recorded 13.39% sand and 80.00% silt, PSA_2509 recorded 45.59% sand and 52.20% silt and PSA_2516 recorded 34.63% sand and 62.96% silt; these differences are considered too large to be due to different laser instrument type alone and are possibly due to how the sample was prepared and/or presented to the laser analyser.

3.2.3.2 Sixty-ninth distribution – PS69

There was generally good agreement for PS69 between the results from the analysis of the benchmark replicates and those from the participating laboratories (see Figure 3). All participants recorded the samples as Gravelly Sand. The percentage gravel ranged from 23.5% (PSA_2510) to 27.1% (PSA_2507) and percentage sand ranged from 71.4% (PSA_2507) to 76.0% (PSA_2515). Percentage mud was generally higher for those participants who used Beckman Coulter laser instruments however PSA_2509 recorded the highest percentage of mud (3.1%) using a Malvern Mastersizer.

Participant PSA_2507 provided sieve data in one phi intervals rather than half-phi which is why their data appears different to other participants. Table 7 in the PS69 report also shows that they appeared to only analyse a sub-sample of the replicate provided as their greater than and less than 1mm weights are significantly lower than other participants and the Benchmark data.

Participants PSA_2504 and PSA_2507 recorded much higher Coarse and less Medium and Fine sand compared to other participants and the Benchmark data (see Table 1 below).

	BM Average	PSA-2504	PSA_2507	Average of remaining participants
% Coarse Sand	26.14	54.04	50.26	27.35
% Medium Sand	44.01	20.85	20.45	43.11
% Fine Sand	4.42	0.02	0.57	4.62

Table 1. Extract of Appendix 2 from PS69, showing percentage Coarse sand, Medium sand and Fine sand recorded by participants.

This could potentially be explained by the use of the Fraunhoffer model that both participants used. The Fraunhoffer approximation is a simplified approach which does not require knowledge of the optical properties of the sample. This can provide accurate results for large particles. However it should be used with caution whenever working with samples which might have particles below 50µm or where the particles are relatively transparent. Participant PSA-2501 does not have a laser analyser and therefore uses an alternate method, although the overall percentage gravel, sand and mud provided matches those following the NMBAQC methodology, Appendix 2 in PS69 Report shows that they recorded less Coarse sand (13.32%) and higher percentage of Medium sand (52.35%) and Fine sand (9.93%).

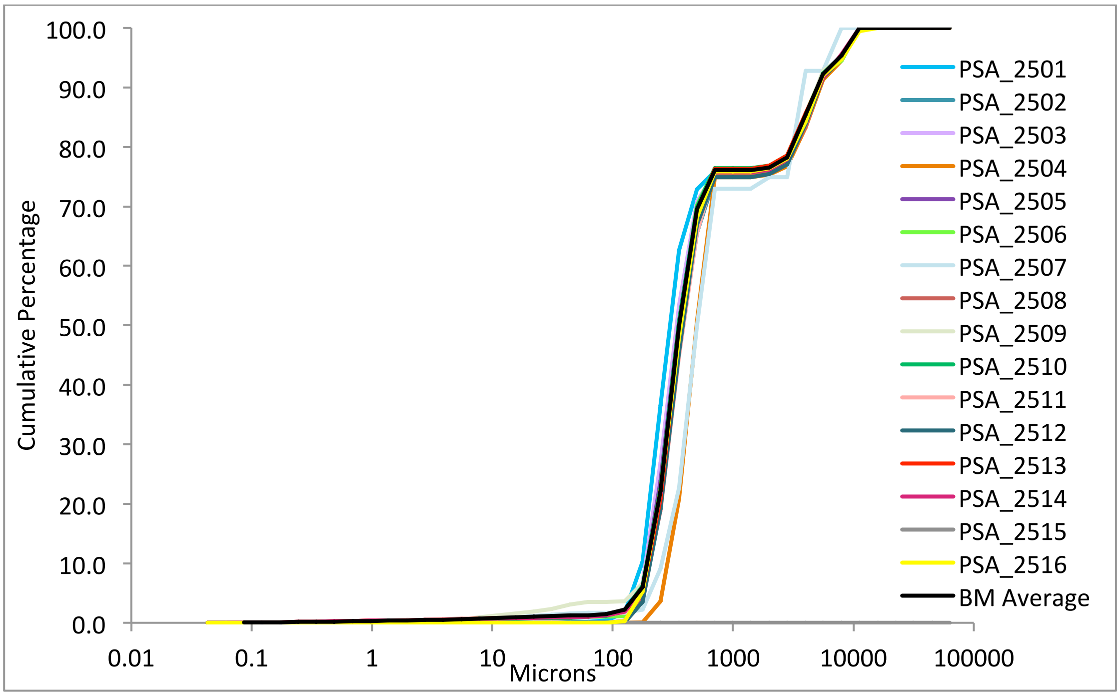

Figure 3. Particle size distribution curves for sediment distributed as PS69 (Figure 6 in PS69).

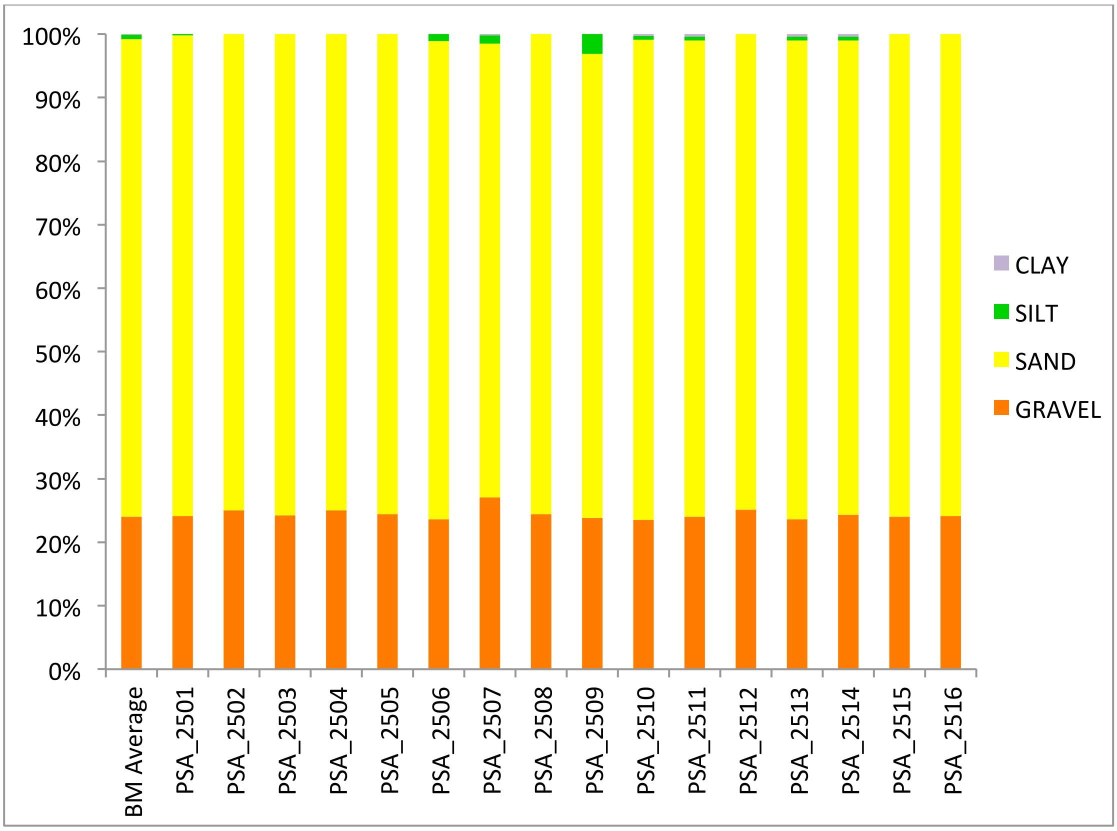

Figure 4. Bar charts showing the percentage gravel, sand, silt and clay for sediment distributed as PS69 (Figure 7 in PS69).

3.2.3.3 Seventieth distribution – PS70

There was generally good agreement for PS70 between the results reported by the participating laboratories and those obtained for the benchmark replicates, as seen in Figure 5; most of the variation occurs below 100microns. All participants had a Gradistat textural group of "Sand" except for PSA_2509 and PSA_2515 who recorded it as "Muddy Sand".

Participant PSA_2501 does not have a laser analyser so followed an alternate Pipette methodology; the main difference between this participant and the Benchmark data is an elevated percentage of Fine sand recorded and a decrease in Coarse sand; this can be seen in Appendix 2 of the PS70 report and in Figure5 below. Of those following the NMBAQC methodology six participants (PSA_2502, PSA_2506, PSA_2508, PSA_2511, PSA_2513, PSA-2514 and PSA_2516) chose to undertake sieve and laser analysis and the remainder only, this had no impact on the percentage gravel as no particles larger than 2mm were found to be present. 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.

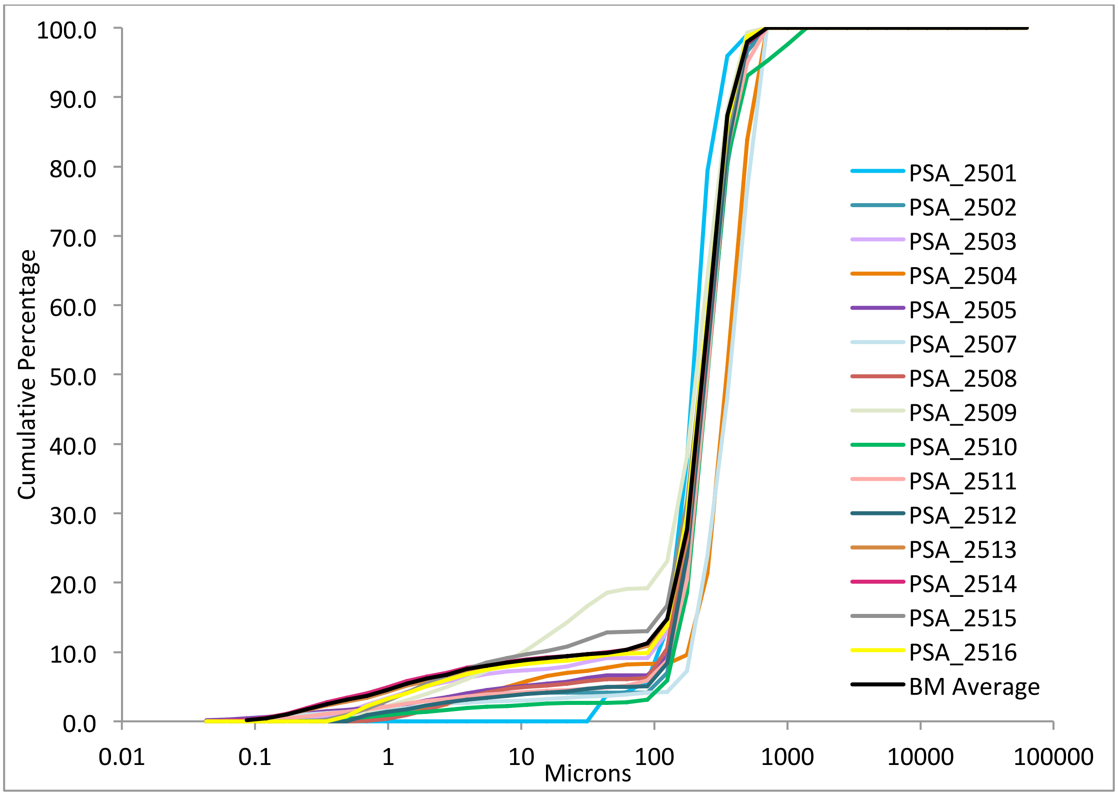

Figure 5. Particle size distribution curves for sediment distributed as PS70 (Figure 6 in PS70).

One participant (PSA_2510) stated that "… small amount of >1 <2mm sediment present but not sufficient to warrant dry sieving >1mm fraction and merging data as [Beckman Coulter] LS 13320 can analyse up to 2mm without issue. This is reflected in the use of laser only data in tabs 4 -7 that includes non-normalised data >707um". Although this is a variation of the NMBAQC method it is valid as the laboratory in question points out the Beckman Coulter laser which is capable of handling larger particles.

Although the overall percentage gravel, sand and mud were similar to other participants, PSA_2504 and PSA_2507 had differing profiles to the Benchmark data and other participants due to differences in the percentage of Coarse sand, Medium sand and Fine sand as shown below in Table 2.

	BM Average	PSA-2504	PSA_2507	Average of remaining participants
% Coarse Sand	12.62	48.95	53.25	14.86
% Medium Sand	59.69	41.51	39.49	58.26
% Fine Sand	16.44	1.21	3.16	17.77

Table 2. Extract of Appendix 2 from PS70, showing percentage Coarse sand, Medium sand and Fine sand recorded by participants.

3.2.3.4 Seventy-first distribution – PS71

There was generally good agreement in results between the laboratories and the benchmark data (see Figure 6). All participants following the NMBAQC methodology used both sieve and laser analysis. Percentage gravel ranged from 34.9% (PSA_2502) to 36.6% (PSA_2504)(Benchmark Average = 35.2%), percentage sand ranged from 54.9% (PSA_2515) to 60.6% (PSA_2507) (Benchmark Average = 57.32%) and percentage mud ranged from 3.48% (PSA_2504) to 9.4% (PSA_2515)(Benchmark Average = 7.49%). Seven participants (PSA_2505, PSA_2509, PSA_2511, PSA_2513, PSA_2514, PSA_2515 and PSA_2516) recorded the sample as "Muddy Sandy Gravel" and the remaining seven participants recorded the sample as "Sandy Gravel".

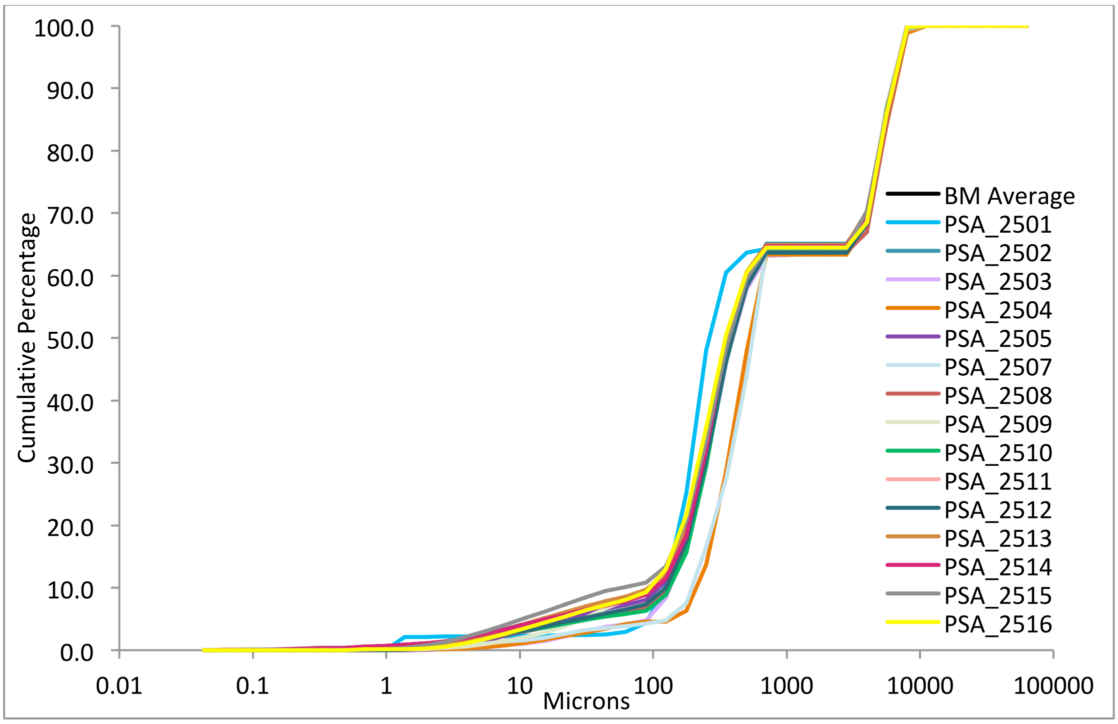

Figure 6. Particle size distribution curves for sediment distributed as PS71 (Figure 6 in PS71).

Figure 6 (and Figure 4 in PS71 Report) shows that the participants have very similar results for the sieve analysis (>1000microns), within the less than 1mm analysis three participants stand out from the rest. Participant PSA_2501 do not have a laser analyser and therefore use an alternate Pipette methodology this resulted in them producing a higher percentage sand and lower percentage mud. Participants PSA_2504 and PSA_2507 appear to be much coarser than the other participants, Table 3 (below) shows that they both recorded much higher percentage coarse sand and lower percentages of medium sand and fine sand compared to the other participants and the Benchmark data.

	BM Average	PSA-2504	PSA_2507	Average of remaining participants
% Coarse Sand	16.21	34.66	36.75	15.13
% Medium Sand	30.30	22.35	19.86	27.28
% Fine Sand	9.07	1.81	3.24	9.53

Table 3. Extract of Appendix 2 from PS71, showing percentage Coarse sand, Medium sand and Fine sand recorded by participants.

Participant PSA_2504 results could potentially be explained by their use of the Fraunhoffer model and use of the “red light only”. The Fraunhoffer approximation is a simplified approach which does not require knowledge of the optical properties of the sample. This can provide accurate results for large particles. However it should be used with caution whenever working with samples which might have particles below 50µm or where the particles are relatively transparent. In Malvern instruments as used by PSA_2504,, light of smaller wavelengths (blue laser) provides improved sensitivity to sub-micron particles, whereas larger wavelengths (red laser) are used to measure larger particles; using the red and blue light combined gives the best resolution and coverage. The NMBAQC recommends using Mie theory as the model with a Particle Refractive index of 1.55 and a Particle Absorption Index of 0.1. Participant PSA_2507 were the only laboratory to use the Fritsch laser analyser and although they stated they used the NMBAQC recommended parameters there are other factors to consider such as laser sub-sample preparation, presentation to the laser, sample homogenisation and the use of ultrasonics.

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_2501 used different methodologies as they do not have access to a laser diffraction instrument. Participant PSA_2510 used a variation of the methodology for exercise PS70 by wet splitting at 2mm, they felt that small amount of >1 <2mm sediment present was not sufficient to warrant dry sieving >1mm fraction and merging data as the [Beckman Coulter] LS 13320 can analyse up to 2mm without issue.

Two of the exercises contained larger quantities of sediment greater than 1mm (PS69 and PS71), the results from the sieve analysis (see Figure 7) show that the processing of sediment greater than undertaken by participants was generally in agreement.

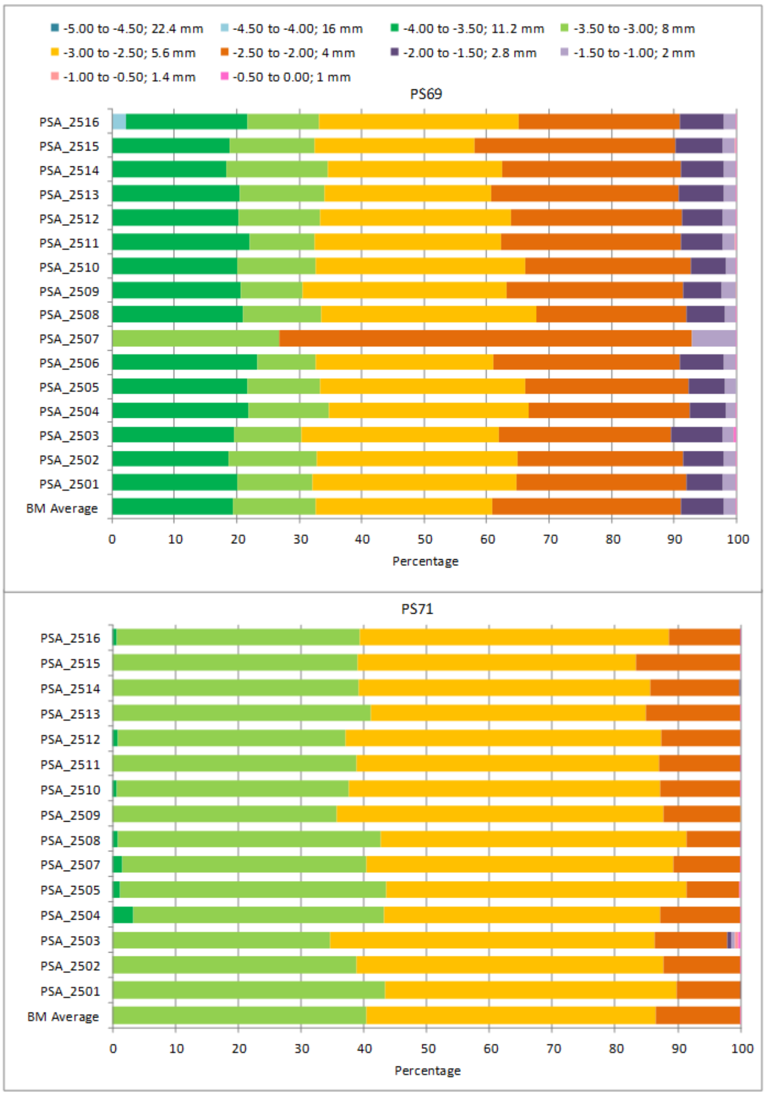

Figure 7. Bar charts showing raw sieve data as percentage in each half-phi interval for PS69 and PS71.

In PS69, participant PSA_2507 provided the greater than 1mm data in phi intervals rather than half-phi intervals which is why it appears different in Figure 7. This participant also appeared to only analyse a sub-sample of the replicate as they only recorded 82.44g of sediment greater than 1mm and 222.22g less than 1mm when the average of the other participants was 242.30g greater than 1mm and 757.49g less than 1mm.

Figure 8 shows the cumulative and differential curves for the laser data for each exercise. Although the results are much improved from previous years laser analysis remains the main causes for concern. 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 hasn't summed to 100% it appears to be due to data entry errors. In exercise PS68, final laser data for participant PSA_2516 sums to 99.98%, this has caused the final merged data not to sum to exactly 100%, however as data is presented to 2 decimal places this has little or no effect on the final distribution. In PS69 the final laser data for participant PSA_2512 sums to 110.37% (Table 8 in PS69 Report) and their data appear displaced in Figure 8 (below) however their laser replicates and final data both sum to exactly 100% and followed a similar distribution to the Benchmark data, it was concluded that the final laser data had been entered into the spreadsheet incorrectly. In exercise PS70, the final laser data for PSA_2507 only sums to 90.62% and does not match the laser replicates provided or the final merged data, again this is considered to be a data entry error into the workbook. For PS71 all participants provided final laser data that had been re-scaled to 100%.

As in previous years it was apparent in the exercises that required laser analysis and had a significant mud fraction (PS69) that there were differences in results depending on which laser instrument was being used. The Coulter instruments have greater measurement sensitivity and were the only instruments capable of detecting particles below 11 phi. The results obtained using the 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. 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.

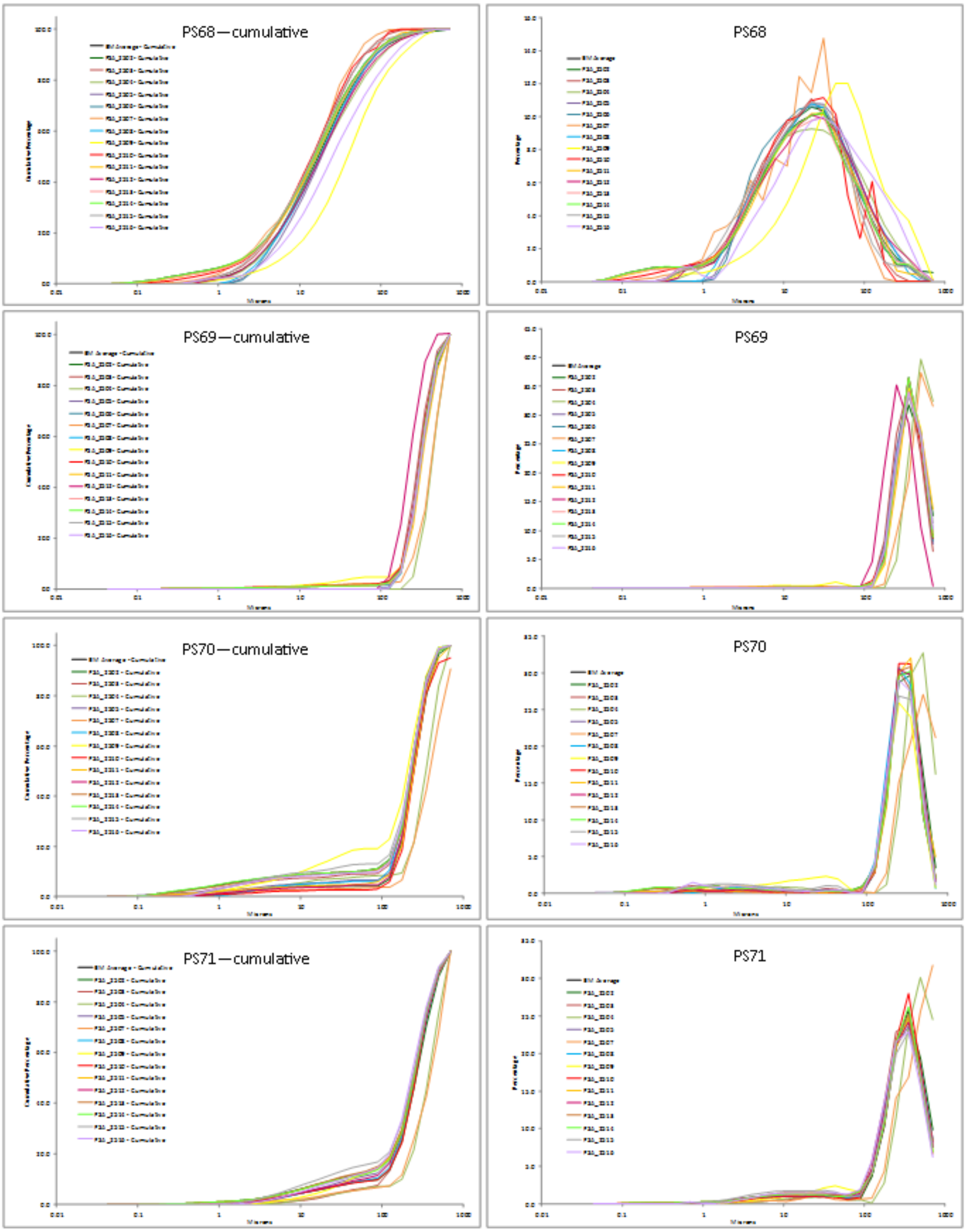

Figure 8. Cumulative and differential final laser data provided by participants for each of the PS exercises.

Laser metadata are very important in helping to identify where possible mistakes are being 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 were updated to make it simpler to provide metadata as participants 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_2503 were the only participant to provide no laser metadata 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 use the NMBAQC Guidance recommendations. Participants that weren't following the recommendations were reminded to do so in their results and some participants changed to the recommended model between exercises. In the first circulation, exercises PS68 and PS69, two participants (PSA_2504 and PSA_2510) used the Fraunhoffer model rather than Mie Theory, one (PSA_2509) used a General Purpose model and PSA_2507 stated, "both" possibly indicating another general purpose model. All participants that provided metadata information used a Particle Absorption Index of 0.1 except for two participants (PSA_2508 and PSA_2516) who used 0.01. Most participants used a Particle Refractive Index of 1.55, variations were 1.45 (PSA_2516), 1.52 (PSA_2509 and PSA_2512) and 1.59 (PSA_2506). 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_2504 who used the red light only. For PS70 and PS71, all participants that provided laser metadata used a Particle Absorption of 0.1, participants PSA_2504 and PSA_2510 still used the Fraunhoffer model and PSA_2509 continued with a General Purpose model. Variations in the Particle Refractive Index still occurred with 1.45 (PSA_2516) and 1.52 (PSA_2509 and PSA_2512). There still 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 high degree of variation in the laser size distributions seen in Figure 8. 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 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 received 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 2018/19 (Scheme year 25) a "Good" or "Review" flag has been issued for methodology and summary data, laser and sieve processing and data merging. This aims to highlight any potential errors but will not be used to assess the performance of a laboratory. 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 is a relatively new module introduced in Scheme year 21 (2014/15) and is a training/ audit module. Participants' "own" samples are re-analysed by the NMBAQC Scheme PSA contractor and the results are compared. The purpose of this exercise was 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 criteria 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, data merging and whether a representative sample was supplied. 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.

4.2 Results

4.2.1 General comments

Twelve laboratories subscribed to the PS-OS module in 2018/19. Two of the eleven labs had multiple lab- codes to facilitate multiple PS-OS submissions due to the sub contraction of samples. All but one participant that subscribed to the module provided data and submitted samples for re-analysis. Participant PSA_2510 had initially signed up but provided email notification of non-participation.

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, data merging, whether a representative sample was supplied and whether the NMBAQC's methodology was being followed. 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 links were provided to information to help resolve problems.

Laboratories generally provided workbooks with all the correct information. All participants except one (PSA_2509) provided all necessary fractions of their sample for re-analysis; participant PSA_2509 did not provide any laser sub-sample, therefore the dried <1mm fractions were used for laser analysis but this required soaking for 48 hours to soften, before thoroughly mixing and subsampling for laser analysis.

There was generally good agreement between the participants and the AQC results, particularly in terms of basic sediment textural classification (see Figure 9).

There were a few discrepancies in the sieve data 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. In some of the results there was a fair amount of variability in the laser analysis between the primary data and the Benchmark re-analysis; some of this variability 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.

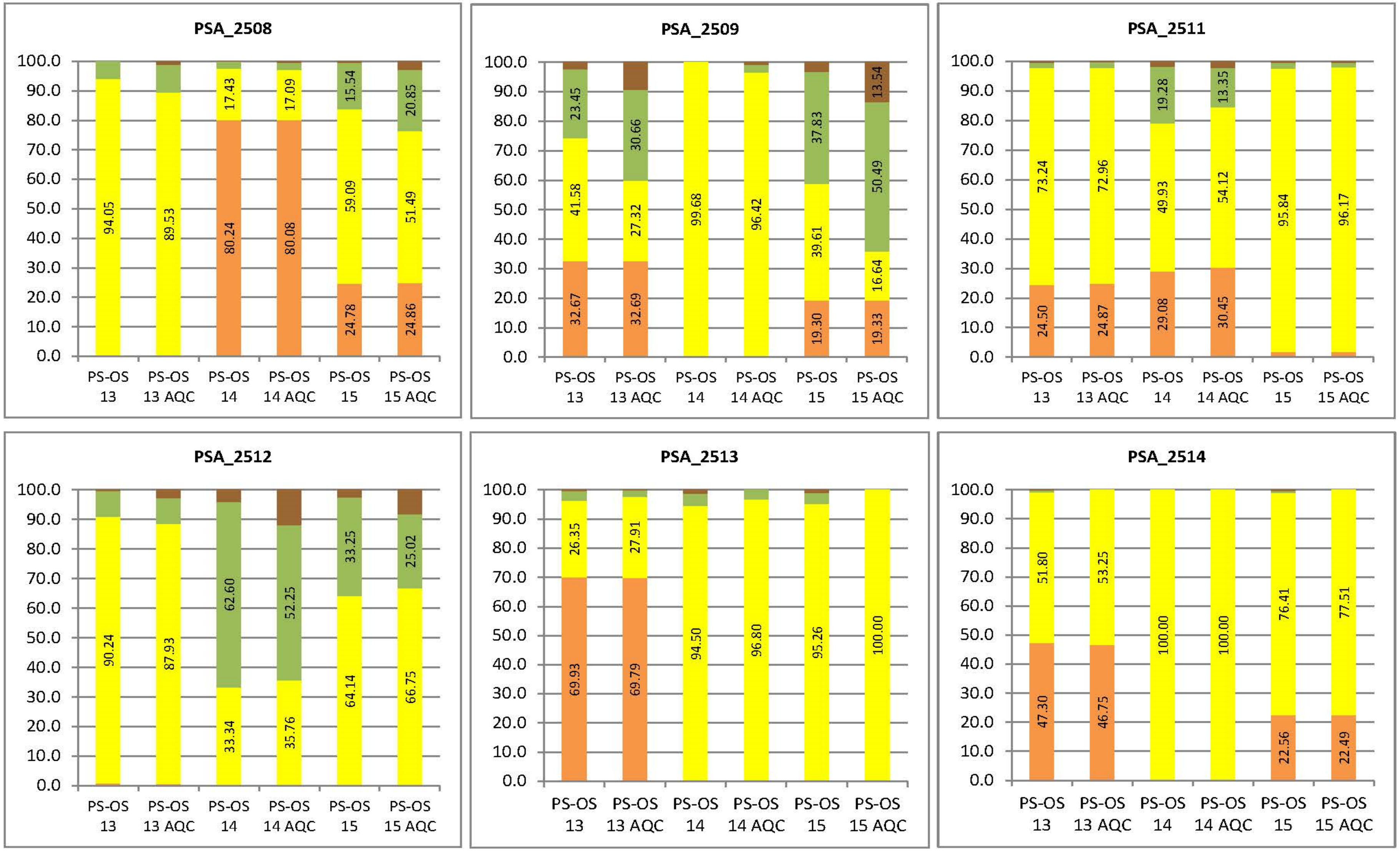

Figure 9.. Bar charts showing percentage gravel, sand, silt and clay from laboratories participating in the PS-OS module.

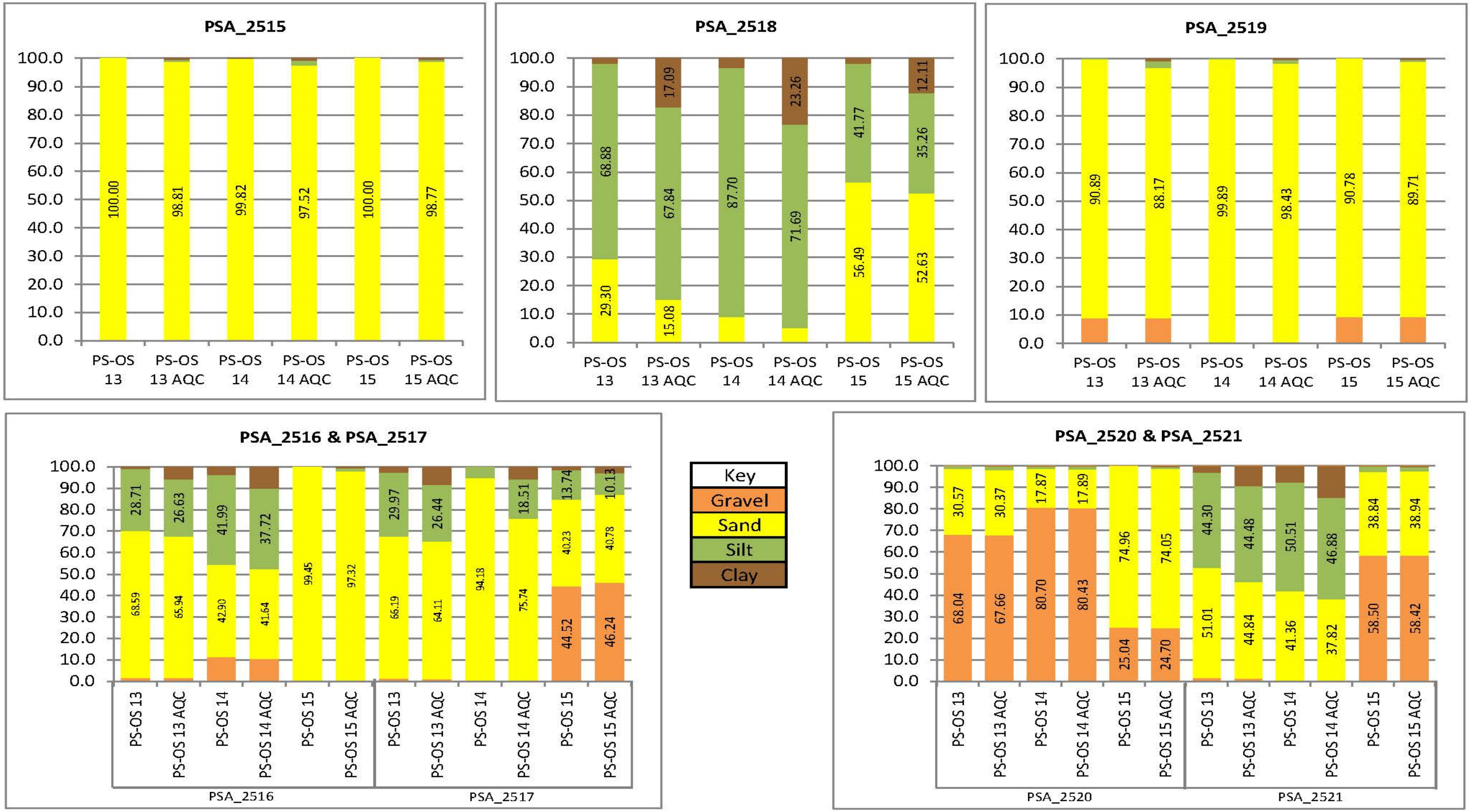

Figure 9. Bar charts showing percentage gravel, sand, silt and clay from laboratories participating in the PS-OS module.

4.3 Discussion

In previous years, the PS-OS module raised issues over the interpretation of the methodology set out in the NMBAQC Best Practice Guidelines (Mason, 2016), in particular how the laser analysis is undertaken. 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. 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 2018/19 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. 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.

3. The current NMBAQC Scheme Pass/Fail criteria for the PS modules are under review. Currently results are broken down for review, including methodology, sieve processing, laser processing, data merging and summary statistics. 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, providing the initial work has been completed this method will be trialled alongside the current “Good” or “Review” format.

4. The PS and PS-OS module results both highlighted differences between the sensitivity of laser instruments. Comparison of laser data in the PS-OS and PS results showed that the Beckman-Coulter LS13320 instrument used by the AQC lab, which includes a

Polarization Intensity Differential Scattering (PIDS) and gives enhanced measurement capability in the clay-size range (<2 um) compared to other lasers models used by many of the NMBAQC scheme participants. The NMBAQC PSA workshop in December 2017 looked at possible ways to minimise the differences created by the use of different laser instruments and optical models, and the possibility of standardising so that all labs following the same procedures. It was agreed that the **recommended optical model is Mie Theory with Particle Refractive index of 1.55 and a Particle Absorption Index of 0.1.** Experimental results have demonstrated that use of the Fraunhoffer optical model reduces the differences between laser instruments, albeit by loss of 'detail' within the very fine silt and clay size fractions. However, the potential suitability of using the Fraunhofer model to achieve greater inter-laboratory comparability will need to be explored in more detail when enough data have been collected. Obscuration will vary depending on sample type; only a small amount of mud is needed to reach an obscuration of 10%, and the presence of relatively small but potentially significant amounts sand may be missed; it may therefore be better to run at a higher obscuration where the presence of sand is observed during sample preparation. A gap can appear between the sieve and laser data in the final merged distribution if not enough sample is added to the laser to detect the sand. **It is essential that participants complete the relevant metadata sections.**

5. **Possible workshop looking at sample preparation and presentation to laser.** 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. A workshop, either in person or a webinar detailing how to create and homogenise a laser sub-sample, particularly looking at the use of ultra0sonics may be useful in forth coming years.

6. **Health and Safety.** Recently the presence of asbestos in marine samples has been bought 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. In light of this, all the natural material used to create PS ring test samples PS68 – 71 was 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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