NMBAQC PSA Questionnaire Summary

A report for discussion at the NMBAQC's Particle Size Analysis for Supporting Biological Analysis Workshop, February 2009

Prue Addison, Environment Agency

1 Background

This report details the results from a questionnaire on Particle Size Analysis (PSA) for supporting biological analysis which was sent out to the Yr 15 participants of the NMBAQC PSA component in June 2008. The participants who provided answers to the questionnaire included the UK's six Competent Monitoring Authorities (CMAs) and six private laboratories that conduct PSA (see Table 1).

Table 1. NMBAQC PSA Participants

Competent Monitoring Authorities
Agri-Food and Biosciences Institute (AFBI)
Centre for Environment, Fisheries & Aquaculture Science (CEFAS)
Environment Agency (EA)
Fisheries Research Services Marine Laboratory (FRS)
Northern Ireland Environment Agency (NIEA)
Scottish Environment Protection Agency (SEPA)
Private Laboratories
AMBIOS
EMU Ltd.
ERT (Scotland) Ltd.
Gardline Environmental
Private Laboratory 1
Private Laboratory 2

2 Questionnaire Aims

The aim of the NMBAQC's PSA Questionnaire is to highlight the current methodological differences in sediment collection, processing and analysis between PSA laboratories in the UK. Through discussions of these methodological differences at the NMBAQC's 'PSA for Biological Analysis' workshop in February 2009, it is the NMBAQC's aim to provide recommendations for key methods which should be included in a UK wide SOP titled 'PSA for Supporting Biological Analysis'.

3 Questionnaire Results

3.1 Sample Collection

All laboratories collect their own PSA samples for supporting biological analysis (including the WFD and CSEMP monitoring programs, in the case of the CMAs), and have written procedures for their methods of collection.

See 1.1-1.3 of the Summary Table for further details.

3.1.1 Minimum Size of PSA Sample Taken

The minimum size of sample collected from a sediment grab varies between the CMAs laboratories and private laboratories (see Table 2).

Table 2. Minimum size of PSA sample taken

Laboratory	Gravel	Sand	Mud
CMAs	50g – 500 mL	50g – 500 mL	25g – 500 mL
Private Laboratories	100g – 800 g	50g – 500 g	25g – 500 g

See 1.4 of the Summary Table for further details.

3.1.2 Source of the PSA Sample

All laboratories take a PSA sub-sample from a day grab. However the purpose (e.g. biology vs chemistry vs separate PSA grab) of the grab varies between laboratories.

Of the CMAs, CEFAS and FRS collect PSA sub-samples from their biology grabs, whilst the EA, NIEA and SEPA collect their PSA sub-samples from separate grabs, and AFBI collect their PSA sub-samples from chemistry grabs (although prior to 2008, they collected from biology grabs).

The private laboratories also vary in their source of PSA sample with two laboratories collecting PSA sub-samples from biology grabs, one laboratory collecting PSA sub-samples from chemistry grabs, and two laboratories collecting PSA sub-samples from separate grabs.

See 1.5 and 1.6a of the Summary Table for further details.

3.1.3 Method of collection of PSA sample

The CMA laboratories method of collection of the PSA sub-sample varies, with most laboratories collecting a depth integrated core/scoop (core dimensions: CEFAS 2 cm diameter, 5 cm deep; FRS 4.5 cm diameter, 1.4 cm deep; SEPA 5 cm diameter, 5-15 cm deep). The volume of the cores taken ranges between 50 g – 500 mL. On the other hand the EA collects a 'metal scoop' of 300-500 mL of sample to 5 cm deep, NIEA collects a 'mixed' sample of 200 mL, and AFBI collects a 250 mL surface sample down to approximately 2 cm deep.

The private laboratories vary in their method of collection of PSA sub-samples, with two laboratories collecting surface sample of volumes 100 and 250 mL; two laboratories collecting a depth integrated core (3 cm and 2-5 cm deep, of volumes 100 g and 500 g respectively) and one laboratory collects a mixed sample between 300-500 mL.

See 1.6 b&c of the Summary Table for further details.

3.1.4 How would you select a sample from a mixed sediment comprising numerous 15cm cobbles set in mud?

The answers to this question given by each laboratory varied substantially: with some laboratories suggesting they would just take a scoop or core directly from an unmixed grab; others suggesting they would mix the sample and then take a sub-sample; others suggested they would take into account the number and size of the cobbles by taking photos and/or descriptions or by measuring a select few cobbles and combining this with the final PSA.

See 1.7 of the Summary Table for each laboratories response to this question.

3.2 Sample Analysis

All laboratories conduct PSA in-house and have written procedures for this except for AFBI, who sub-contract out this work to University of Plymouth, Geography Department (who do have written procedures for PSA analysis). All laboratories use Malvern Lasersizer instruments (except for one private laboratory which uses a pipette and microscope method) and a set of sieves for PSA.

See 2.1-2.5 of the Summary Table for further details.

3.2.1 Drying of PSA samples

Each CMA varies in their approach to drying of PSA samples (despite the Green Book stating that PSA samples should be freeze dried on reception). AFBI, CEFAS, FRS, NIEA and SEPA all freeze their PSA samples, whilst the EA do not freeze dry their samples.

The private laboratories also vary in their approach to drying of PSA samples, with only two laboratories freezing their PSA samples, two laboratories not freezing and two laboratories oven drying samples.

See 2.6 of the Summary Table for further details.

3.2.2 The use of hydrogen peroxide to remove organic material

Of the CMA laboratories, only AFBI uses hydrogen peroxide to remove organic material. Only three of the five private laboratories use hydrogen peroxide to remove organic material.

See 2.7 of the Summary Table for further details.

3.2.3 PSA sub-sample collection

All laboratories homogenise their samples by mixing with a spatula or by mixing/inversion of the sample container.

The CMA laboratories either use a spatula (AFBI, CEFAS, EA), plastic scoop (FRS, SEPA) or sediment riffles (NIEA) to obtain a sub-sample for sediment analysis. The private laboratories use either a plastic scoop or spatula to obtain a sub-sample or use their whole sample (with a portion for sieving and a portion for laser/pipette analysis).

The range in volume of sub-sample analysed for different sediment types is outlined in Table 3.

Table 3. Minimum volume of sub-sample analysed

Laboratory	Gravel	Sand	Mud
CMAs	100 g – whole sample	0.1 g - whole sample	0.1 – 25 g
Private Laboratories	200 – 800 g	4 – 250 g	1.5 – 200 g

See 2.8 – 2.10 of the Summary Table for further details.

3.2.4 Are live/dead fauna/shells removed?

All CMA laboratories remove live/dead fauna/shells prior to sediment analysis. However, only CEFAS record the weight and fraction that the fauna/shells are present in, and also complete identification of the fauna/shell if possible.

Most private laboratories remove live/dead fauna/shells prior to sediment analysis, except for one which only removes seaweed.

See 2.11 – 2.12 of the Summary Table for further details.

3.3 Processing a sample

3.3.1 Is a dispersant (e.g. sodium hexametaphosphate) used?

Of the CMA laboratories, only NIEA use a dispersant to separate cohesive/consolidated particles. No other CMA laboratories use a dispersant during laser analysis.

Of the six private laboratories, one uses a dispersant for processing a sample, whilst three of the other laboratories explain that they would use a dispersant if a sample was significantly cohesive/consolidated, whilst the other two laboratories do not use a dispersant.

See 2.17 of the Summary Table for further details.

3.3.2 What Obscuration for laser analysis is specified in SOP?

The Obscuration ranges detailed in each laboratories SOPs vary between 5-30 %.

Most of the CMA laboratories maximum Obscuration values fall within the suggested range of 10-20 % by Malvern Instruments, except for AFBI who conducts laser analysis up to 25% Obscuration. Three of the five private laboratories have maximum Obscuration's above Malvern Instruments suggested 20 %.

See 2.18 of the Summary Table for further details.

3.3.3 Laser and Sieve Fractions

For the CMA laboratories, the method of separating the laser fraction from the sieve fraction varies with AFBI, CEFAS and EA wet sieving (at 0 phi, 4 phi and -1 phi respectively) and FRS and NIEA dry sieving (at -1 phi and 0 phi respectively), whilst SEPA does not sieve at this stage of the process and merely take a sub sample of their entire sample and put it into the lasersizer.

Sieve analysis also varies, with all laboratories dry sieving except for EA who wet sieve. The minimum sieve sizes used (and therefore maximum laser size) ranges from -1 to 4 phi. Both AFBI and CEFAS conduct their sieve analysis at ½ phi intervals, whilst the EA, FRS, NIEA and SEPA all conduct their sieve analysis at 1 phi intervals (each of these laboratories have suggested they would move to ½ phi intervals if needed). All CMA laboratories conduct their laser analysis at ½ phi intervals.

For the private laboratories, the method of separating the laser fraction from the sieve fraction varies with three laboratories dry sieving (to -1 phi and 1 phi), and three wet sieving (to 4 phi and 0 phi).

Sieve analysis also varies, with only one laboratory wet sieving and the other five dry sieving. The minimum sieve size (and therefore maximum laser size) ranges from -1 to 4 phi. Three of the laboratories conduct their sieve analysis at ½ phi intervals whilst the others use 1 phi intervals. All of the laboratories conduct their laser analysis at ½ phi intervals.

See 2.19 in the Summary Table for further details.

3.4 Data Interpretation, Reporting and Storage

All laboratories are responsible for the interpretation, reporting or storage of particle size data.

Of the CMA laboratories, the EA and FRS use the Malvern Software to merge their sieve and laser data and calculate the derived statistics, whilst AFBI, CEFAS, NIEA and SEPA all use their own spreadsheets (AFBI also uses the program Gravistat in conjunction with their own spreadsheet).

Of the private laboratories one uses the Malvern Software to merge their sieve and laser data and calculate the derived statistics, whilst the three laboratories use their own spreadsheets, and one laboratory uses the program Gravistat.

See 3.1-2 in the Summary table for further details.

3.4.1 Derived statistics and fractions reported

There is variation in the derived statistics reported by the CMAs and private laboratories with most laboratories reporting Folk and Wards (1957) Inclusive Graphic Statistics, however some reporting Dyer (1986) or Pettijohn's (1973) Method of Moments statistics (see Table 4).

Table 4. Derived statistics reported by the CMAs

	Mean	Median	Sorting	Skewness	Kurtosis
CMAs					
AFBI	Inclusive	ϕ50	Inclusive	Inclusive	Inclusive
CEFAS	Moments	ϕ50	Moments	-	Moments
EA	Inclusive	ϕ50	Inclusive	Inclusive	Inclusive
FRS	Inclusive	ϕ50	Inclusive	Inclusive	Inclusive
NIEA	Inclusive	ϕ50	Inclusive	Inclusive	Inclusive
SEPA	$M_\Phi = (\Phi_{16} + \Phi_{84})/2$	ϕ50	Inclusive	Inclusive	Inclusive
Private Laboratories					
AMBIOS	Inclusive or Moments	Inclusive or Moments	Inclusive or Moments	Inclusive or Moments	Inclusive or Moments
EMU Ltd.	Inclusive	ϕ50	Inclusive	Inclusive	Inclusive
ERT (Scotland)	Moments	-	Moments	Moments	-
Gardline	Inclusive	ϕ50	Inclusive	Inclusive	Inclusive
Private Laboratory 1	Inclusive	-	Inclusive	Inclusive	Inclusive
Private Laboratory 2	Inclusive	ϕ50	Inclusive	Inclusive	-

All laboratories that report the silt/clay, sand and gravel fractions use the phi ranges of >4 phi, -1 to 4 phi and < -1 phi respectively.

See 3.3 in the Summary table for further details.

3.4.2 How are these data stored?

All laboratories have raw data and the derived stats held either in a database or spreadsheet, and for some laboratories these are also kept as hardcopies.

See 3.4 in the Summary Table for further details.

3.5 Quality Control / Quality Assurance

3.5.1 QA/QC procedures

Each laboratory varies in the amount of in-house QA/QC conducted. Generally most laboratories have equipment checks done (e.g. servicing of Malvern Laser, check of laser with reference material, daily check of balance, balance checked with certified weights), however very few have an internal QA check of re-analysis of a certain number of samples (which includes re-analysing both the laser and sieve fraction). The only laboratories that do this are NIEA, SEPA and one of the private laboratories.

See 4.1 in the Summary Table for further details.

3.5.2 Comments about the NMBAQC's PS module

All laboratories which have participated in the NMBAQC's Particle Size Module, have provided the following feedback:

- Useful dataset, needs good sedimentologist to analyse.
- Should be more samples with a wider mix of sediment types.
- A more thorough interpretation of results would be beneficial, incld representation of all raw data from participating groups, and not just derived stats. This may allow participating groups that fail to pin point where they may be making errors (whether during analysis or data interpretation).
- We normally report our routine results in μm not phi units.
- The PS module is very good at indicating any problems with outputs and methodologies.

See 4.2-4.3 in the Summary Table for further details.

4 Concluding remarks

The following points outline the areas of methodological differences which we suggest are the most important aspects of PSA that need to be standardised in order to improve both the quality and comparability of data produced by different laboratories:

Sample Collection

- Source of PSA sub-sample (biology vs separate grab).
- Method of sub-sample collection (depth integrated core/mixed sample/surface sample) and the volume collected.

Sample Analysis

- Sample preservation (Freezing/not freezing/oven drying).
- Removal of organic material with hydrogen peroxide vs. no removal of organic material.
- Removal of conspicuous fauna (i.e. snail shells, urchins, etc.) vs. no removal of fauna.
- Volume of sub-sample used for laser and sieve analysis.
- Obscuration range of laser analysis.
- The use of a dispersant vs. no dispersant.
- Wet/Dry sieving (to what size) to separate laser and sieve fraction.

Data Interpretation, Reporting and Storage

- Calculating derived statistics via Malvern Software vs. Own Spreadsheets.
- The derived stats reported (Inclusive vs Moments).

QA/QC

- The varying levels of internal QA/QC done by laboratories.