

The National Marine Biological Analytical Quality Control Scheme

Particle Size Analysis Component
Report from the Contractor
Scheme Operation – Year 17
2010/11

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PARTICLE SIZE ANALYSIS COMPONENT REPORT FROM THE CONTRACTOR

SCHEME OPERATION - YEAR 17 - 2010/11

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Year 17 Exercise Reports (hyperlinked in this report)

Particle Size Results - PS36

Particle Size Results - PS37

Particle Size Results - PS38

Particle Size Results - PS39

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1.1

The National Marine Biological Analytical Quality Control (NMBAQC) Scheme addresses three main areas relating to benthic biological data collection:
 □ The processing of macrobenthic samples. □ The identification of macrofauna. □ The determination of physical parameters of sediments.
The seventeenth year of the Scheme (2010/11) followed the format of the sixteenth year. A series of exercises involved the distribution of test materials to participating laboratories and the centralised examination of returned data and samples. The labelling and distribution procedures employed previously have been maintained and specific details can be found in the Scheme's annual reports for 1994/95 and 1995/96 (Unicomarine, 1995 & 1996).
In the Year 17 NMBAQC Scheme twelve laboratories participated in the particle size analysis components PS36 and PS37; five were government laboratories; seven were private consultancies. Five of the participants were responsible for CSEMP (Clean Seas Environment Monitoring Programme) sample analysis. Nine laboratories participated in components PS38 and PS39; five were government laboratories; four were private consultancies. Five of the participants were responsible for CSEMP sample analysis. To reduce potential errors and simplify administration, LabCodes were assigned in a single series for all laboratories participating in the benthic invertebrates, fish and particle size components of the NMBAQC Scheme (due to Thomson Unicomarine administering these three components).
As in previous years, some laboratories elected to be involved in limited aspects of the Scheme. CSEMP laboratories were required to participate in all relevant components of the Scheme, although this was not strictly enforced.
Summary of Performance
This report presents the findings of the Particle Size Analysis components for the seventeenth year of operation of the National Marine Biological Analytical Quality Control (NMBAQC Scheme.
This component consisted of one module with four exercises:

 $\hfill\square$ Analysis of four sediment samples for physical description (Particle Size module).

The analytical procedures of this module were the same as for the sixteenth year of the Scheme. The results for the four exercises are presented and discussed. Comments are provided on the performance for each of the participating laboratories in each of the exercises.

In previous years the Particle Size exercises (PS) 'Pass/ fail' criteria were based upon z-scores from the major derived statistics with an acceptable range of ±2 standard deviations (see Description of the Scheme Standards for the Particle Size Analysis Component). The annual report for Scheme Year 16 deemed the use of z-scores inappropriate for such a low number of data returns where two erroneous results can significantly alter the pass / fail criteria. The z-score method also assumes that the majority of respondents are correct and raised genuine concerns regarding technique and method bias. Following this, the 'Pass/ fail' criteria are currently under review and an alternative flagging criteria is being trialled in Scheme Year 17. The seventeenth year is trialling the use of z-scores calculated for each half-phi interval.

The variation within the ten *replicate* results produced by the benchmark laboratories using the NMBAQC PSA SOP was minimal for PS36-39; this is partly attributable to the use of only Malvern laser instruments and some standardised protocols, *i.e.* no use of chemical dispersants or hydrogen-peroxide pre-treatment. In most cases there was reasonably good agreement between participant laboratories for all four PS exercises. The first particle size exercise of the Scheme year (PS36; sandy mud sample) received twelve data returns. The second particle size exercise of the Scheme year (PS37; sand sample) received twelve data returns. The third particle size of the Scheme year (PS38; artificial gravel sample) received nine data returns. The final particle size exercise of the Scheme year (PS39; gravelly muddy sand sample) received nine data returns.

Comments are provided on the individual performance of the participating laboratories in each of the above components. A summary of their performance with respect to standards determined for the CSEMP is presented.

1.1.1.1 Statement of Performance

Each participating laboratory received a 'Statement of Performance', which included a summary of results for each of the Schemes modules and details the resulting flags where appropriate. These statements were first circulated with the 1998/1999 annual report, for the purpose of providing proof of Scheme participation and for ease of comparing year on year progress.

2. Summary of PSA Component

2.1 Introduction

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

This module is described in more detail below. A brief outline of the information to be obtained from the module is given, together with a description of the preparation of the necessary materials and brief details of the processing instructions given to each of the participating laboratories.

2.1.1 Logistics

The labelling and distribution procedures employed previously have been maintained and specific details can be found in the Scheme's annual reports for 1994/95 and 1995/96 (Unicomarine, 1995 & 1996). Email was the primary means of communication for all participating laboratories. This has considerably reduced the amount of paper required for the administration of the Scheme.

2.1.2 Data returns

Return of data to Thomson Unicomarine Ltd. followed the same process as in previous years. Spreadsheet based forms (tailored to the receiving laboratory) were distributed for each circulation via email, with additional hard copies where appropriate. All returned data have been converted to Excel 2003 format for storage and analysis. In this and previous Scheme years slow or missing returns for exercises lead to delays in processing the data and resulted in difficulties with reporting and rapid feedback of results to laboratories. Reminders were distributed shortly before each exercise deadline.

2.1.3 Confidentiality

To preserve the confidentiality of participating laboratories, each are identified by a four-digit Laboratory Code. In September 2010 each participant was given a confidential, randomly assigned Scheme year seventeen LabCode. Codes are prefixed with the Scheme year to reduce the possibility of obsolete codes being used inadvertently by laboratories, *e.g.* Laboratory number four in Scheme year seventeen will be recorded as LB1704.

In this report all references to Laboratory Codes are the post-August 2010 codes (Scheme year seventeen). To reduce potential errors and simplify administration, LabCodes were assigned in a single series for all laboratories participating in the benthic invertebrate, fish and particle size components of the NMBAQC Scheme (due to Thomson Unicomarine administering these three components).

2.2 Particle Size Analysis (PS) Module

2.2.1 Description

This component examined the percentage of sediment found in each half-phi interval from the particle size analysis of replicate sediment samples. Four samples of sediment, one fine (PS36), two coarser (PS37 and PS38) and one mixed (PS39) were distributed in 2010/11. The sets of PS36 and PS37 replicate samples were derived from natural marine sediments; PS38 replicates were artificially prepared from commercial aggregate materials; PS39 replicates were prepared from combined natural sediments; they were prepared as described below. In each case a random subsample of the prepared replicates were divided for laser diffraction analysis to ensure sample replicate consistency. For PS36 - 39 the *replicates* were analysed using a Malvern Mastersizer 2000 to produce benchmark data.

2.2.1.1 Preparation of the Samples

The first two PS circulations were sediments collected from natural marine environments (Harwich for PS36, Nobel Bank for PS37); the third (PS38) was artificially created from commercially acquired materials; the final sediment (PS39) was artificially created from combined natural sediments (Harwich and the English Channel) and commercially acquired materials. Natural material for PS36 was returned to the laboratory and coarse sieved (1 mm) to remove gravel, shell and large faunal content. A minimum of 30 litres of visually similar sediment was collected. Following sieving, the sediment for PS circulation was well mixed in a large tray and allowed to settle for a week. The sediment was sub-sampled by coring in pairs. One core of a pair was stored as the 'A' component, the other as the 'B'. To ensure sufficient weight for analysis, and to further reduce variation between distributed PS samples, this process was repeated three times for each sample replicate, i.e. each distributed sample was a composite of three cores. The artificial PS38 replicates were produced by combining known quantities of commercially acquired material. For the PS39 replicates, subtidal shell sands from the English Channel were passed through the sieve shaker and known weights from each half-phi interval were added to known quantities of mud from PS36 (Harwich), also added was a known weight of commercial pebble.

The numbering of the replicate samples was random. All of the odd-numbered 'B' components (a total of 14) were sent for particle size analysis to assess the degree of inter-sample variation and produce benchmark data. All of these *replicates* were analysed using a Malvern Mastersizer 2000 laser. The 'A' components were assigned to participating laboratories randomly and distributed according to the Scheme timetable.

2.2.1.2 Analysis required

The participating laboratories were required to conduct particle size analysis on the samples following the NMBAQC's best practice guidance for particle size analysis to support biological data (Mason, 2011), either in-house or using a subcontractor. A written description of the sediment characteristics was to be recorded (pre-processing and post-processing using the Folk Triangle) as well as the % <63µm and an indication of any peroxide treatment or chemical dispersant used. Also requested was a breakdown of the particle size distribution of the sediment, to be expressed as a weight or percentage of sediment in half-phi (\$\phi\$) intervals. Optional data on the mean, median, sorting and skewness from the GRADISTAT program could also be provided. Approximately **nine weeks** were allowed for the analysis of each PS sample (PS36, PS37, PS38 and PS39).

2.2.2 Results

2.2.2.1 General comments

Thirteen laboratories subscribed to the exercises in 2010/11. One of the laboratories did not submit returns for any of the exercises and three did not submit returns for the second two exercises (PS38 and PS39).

Most participating laboratories now provide data in the requested format, though some variations remain. As previously reported, it should be remembered that the results presented may be from a more limited number of analytical laboratories than is immediately apparent since this component of the Scheme is often sub-contracted by participants to one of a limited number of specialist laboratories. For PS36, twelve out of thirteen participating laboratories returned data; one laboratory did not provide data or provide notification of abstention. For PS37, twelve out of thirteen participating laboratories returned data; one laboratory did not provide data or provide notification of abstention. For PS38, nine out of thirteen participating laboratories returned data; four laboratories did not provide data, three of which did not provide notification of abstention. For PS39, nine out of thirteen participating laboratories returned data; four laboratories did not provide data, three of which did not provide notification of abstention. Detailed results for each exercise have been reported to the participating laboratories (PS36, PS37, PS38 and PS39); additional comments are added below.

2.2.2.2 Analysis of sample replicates (benchmark data)

Replicate samples of the sediment used for the four PS distributions were analysed where required using a Malvern Mastersizer 2000 with Hydro-G Dispersion unit (no blue laser) to examine replicate variability and establish benchmark data. Replicates have been examined by both laser and sieve / pipette methods in earlier Scheme years; however as the majority of

laboratories are conducting analyses by laser diffraction the testing of *replicates* is now undertaken using laser instruments. In Year 16, half the *replicates* were analysed using a Malvern Mastersizer 2000 laser and half by a Malvern Mastersizer X log bed laser. In Year 17 *replicate* analyses were performed by Plymouth University, Geography Department (Malvern Mastersizer 2000) (PS36 and PS37) and Thomson Unicomarine Ltd (Malvern Mastersizer 2000) (PS38 and PS39). *Replicate* samples analysed by both laboratories showed very good agreement.

Sample PS36 comprised of sandy mud sediment (average of 54.53% <63µm, mean phi of 4.76), the Malvern Mastersizer 2000 showed no discernable variation between *replicate* samples. Results for the individual *replicates* are provided in <u>Table 1</u> and are displayed in Figure 1 (PS36 Report).

Sample PS37 comprised a sand sediment (average of 0.00% <63µm, mean phi of 1.53). The Malvern Mastersizer 2000 showed good agreement between *replicate* samples. Results for the individual *replicates* are provided in Table 1 and are displayed in Figure 1 (PS37 Report).

Sample PS38 comprised of an artificial gravel sediment (average of $0.00\% < 63\mu m$, mean phi of -2.96). The replicates were analysed by dry sieving only, no laser analysis was required. Results for the individual *replicates* are provided in <u>Table 1</u> and are displayed in <u>Figure 1</u> (PS38 Report).

Sample PS39 of Gravelly Muddy Sand sediment (average of 35.63% <63µm, mean phi of 3.00). Replicates showed no discernable variation, results for the individual replicates are provided in Table 1 and are displayed in Figure 1 (PS39 Report).

2.2.2.3 Results from participating laboratories

Where they were provided summary statistics for the four PS circulations are presented in Table 1 in each individual exercise report (see PS36 Report, PS37 Report, PS37 Report, PS38 Report, PS38 Report, PS37 Report, PS37 Report, PS38 Report, PS39 Report, PS39 Report, PS36 Report, PS37 Report, PS36 Report, PS37 Report, PS39 Report, PS39 Report) shows a summary of the z-scores calculated for each half phi interval. Intervals left blank or marked "not analysed" were entered as zero to calculate the z-scores. Each individual exercise report also provides a bar-chart of each lab's z-scores.

2.2.2.4 Thirty-sixth distribution - PS36

There was generally good agreement for PS36 between the results from the analysis of *replicates* and those from the majority of participating laboratories (see Figure 2). One laboratory (LB1703) pre-treated their replicate with hydrogen peroxide; this is likely to be accountable for their high % silt / clay value (75.63%) and the displacement of their cumulative curve. One lab (LB1703) provided data in a different format to the half-phi intervals requested and LB1728 had missing data values for some of the half-phi intervals towards the end of the data set. All of the participants used the laser diffraction technique to analyse the sample. Table 1 shows the variation in data received from the participating laboratories. The derived statistic for %silt/clay ranged from 48.18% to 75.63%, excluding data from the *replicate* analyses produced by Plymouth University, Geography Department (Malvern Mastersizer 2000).

2.2.2.5 Thirty-seventh distribution - PS37

There was generally good agreement for PS37 between the results from the analysis of *replicates* and those from the participating laboratories (see Figure 2). One laboratory (LB1703) pre-treated their replicate with hydrogen peroxide; although this did not appear to have any effect on their results. Four laboratories (LB17012, LB1713, LB1716 and LB1726) stated that they used laser diffraction only to analyse the sample; seven laboratories (LB1701, LB1702, LB1703 LB1705, LB1707, LB1714 and LB1728) used a combination of sieving and laser diffraction to analyse the sample; and one participant (LB1715) dry sieved their entire sample. Table 1 shows the variation in data received from the participating laboratories. The derived statistic for %silt/clay ranged from 0.00% to 1.29%, excluding data from the *replicate* analyses produced by Plymouth University, Geography Department (Malvern Mastersizer 2000).

2.2.2.6 Thirty-eighth distribution - PS38

There was very good agreement for PS38 between the results from the analysis of *replicates* and those from the participating laboratories (see Figure 2). Most laboratories (LB1701, LB1702, LB1707, LB1712, LB1713, LB1715, LB1716 and LB1726) only used dry sieving to analyse the sample. One lab (LB1705) attempted laser diffraction as well as dry sieving but found there was insufficient sediment to do more than one run through the laser. Four labs (LB1712, LB1715, LB1716 and LB1726) did not provide the data in half phi intervals. One lab (LB1712) provided data on a completely different scale. Table 1 shows the variation in data received from the participating laboratories where data was submitted. The derived statistic for the % silt/clay was 0% for all laboratories.

2.2.2.7 Thirty-ninth distribution - PS39

There was a fair amount of variation between the results from analysis of replicates and those from the participating laboratories (see Figure 2). Eight laboratories (LB1701, LB1702, LB1705, LB1707, LB1712, LB1713, LB1716 and LB1726) used sieve and laser analysis to analyse the sample; one lab (LB1715) only used laser analysis. LB1705 didn't analyse below - 3.5φ, displacing their cumulative curve by 1 phi at the beginning. LB1715 only used laser analysis and therefore did not record any material below -1.0φ. The derived statistic for the % silt/clay ranged from 13.20% to 52.06%, excluding data from the *replicate* analyses produced by Thomson Unicomarine (Malvern Mastersizer 2000).

2.2.3 Discussion

The samples distributed as PS36 appeared from an analysis of *replicates* (Figure 1) to be good replicates with very little variance. Results from participating laboratories (Figure 2) showed a general similarity in distribution curves, except for that of LB1703; who provided data in a different format and used in-house methods rather than following the NMBAQC Scheme's Standard Operating Procedure. This was the only laboratory to pre-treat the sample with hydrogen peroxide, which resulted in a %silt/clay value 75.63%, higher than the average. One laboratory (LB1728) recorded sediment present between -0.5\phi and 0.5\phi which no other laboratory recorded: this accounts for the high z-score received between -0.5\phi and 1.0\phi. LB1726 and LB1707 recorded slightly than average data between 1.5\phi to 5.5\phi and 4.0\phi to 7.5 \phi respectively; this accounts for the higher z-scores received by these labs between these phi intervals. LB1701 received a high z-score for the -1.5\phi to 1.0\phi interval; this is due to their being the only laboratory to record data in this interval. This is where the z-score method of analysing is inappropriate because it does not take into consideration that the differences are minimal.

The samples distributed as PS37 appeared from an analysis of *replicates* (Figure 1) to be good replicates with little variance. Results from participating laboratories were concurrent (Figure 2). Three of the participating laboratories (LB1701, LB1705 and LB1714) stated that the only equipment used to analyse the sample was a laser when all three laboratories have in fact provided data in the "Final Sieve" tab in the workbook. One laboratory's (LB1726) cumulative percentage curved is displaced from the other cumulative percentage curves (Figure 2) which appears to be because the data has been submitted half a phi-interval out. Despite the cumulative percentage curves following a very similar shape, the z-score analysis still produces a few high values. As in PS36, the z-score analysis appears inappropriate because high z-scores are being calculated when the difference between laboratories, in some cases is only a tenth of a gram.

The samples distributed as PS38 appeared from an analysis of *replicates* (Figure 1) to be good replicates with little variance. Results from participating laboratories were in accord (Figure 2). Four of the participating laboratories (LB1712, LB1715, LB1716 and LB1726) did not provide data in half-phi intervals, this accounts for the deviation in their cumulative percentage curves (Figure 2). Three of these labs (LB1715, LB1716 and LB1726) did state "-" (not analysed) for these half phi intervals rather no data recorded. LB1712 provided data in a different format, this data set was modified so it could be directly compared to the other participating laboratories. This may account for the slight deviation in the LB1712 cumulative percentage curve between -4.0 φ and -3.5 φ . The lab that attempted laser analysis (LB1705) received high z-scores from 4.0 φ and above because they were the only lab to record data in these half-phi intervals. As with PS36 and PS37, a lot of the high z-scores are caused by small deviations.

The samples distributed as PS39 appeared from an analysis of replicates (Figure 1) to be good replicates with little variance. Results from participating laboratories showed a high level of variation (Figure 2). Eight of the participating laboratories (LB1701, LB1702, LB1705, LB1707, LB1712, LB1713, LB1716 and LB1726) used sieves and laser diffraction to analyse the sample, however three of these laboratories (LB1701, LB1705 and LB1726) stated that the only equipment used to analyse the sample was a laser when in fact they have all provided sieve data. One laboratory (LB1715) only used a Coulter Laser Granulometer to analyse the sample. One laboratory (LB1705) did not analyse below -3.5¢, therefore the start of their cumulative percentage curve has been displaced by one phi. This also accounts for the high zscore they received for the -3.5\psi to -3.0\psi interval. The lab that only used laser diffraction (LB1715) did not record any material below -1.0\(\phi\). Presumably this laboratory decided to ignore the pebble component supplied in their replicates. One laboratory (LB1701) failed to record the pebble component despite analysing the sample using sieves and laser diffraction; this appears not to be an omission due to policy as zeros are entered in their final data sheet. The majority of variation within this PS exercise appears after 0.0.¢, when laser diffraction is employed.

Participating laboratories were asked to provide a visual description of the PS36, PS37, PS38 and PS39 samples prior to analysis and instructed to describe the sediment using the Folk triangle post analysis. Data were provided by all twelve participating laboratories for PS36 and PS37 and by all nine participating laboratories for PS38 and PS39. For PS36, prior to analysis the majority of labs (7) (LB1701, LB1702, LB1707, LB1712, LB1714, LB1715 and LB1728) recorded Sandy Mud, one (LB1726) recorded Fine Sandy Mud, three (LB1703, LB1705 and LB1713) recorded Mud and one lab (LB1716) recorded Silt. Post analysis of PS36, eight laboratories recorded Sandy Mud and three (LB1712, LB1715 and LB1716) recorded varying degrees of silt. For pre and post analysis of PS37 all laboratories recorded varying accounts of Sand. For PS38, all laboratories recorded Gravel for pre and post analysis, some labs gave a

more in-depth description for their pre analysis characterisation. For PS39 most laboratories concluded post-analysis that the sample was gravelly Muddy Sand ((g) mS). There was some variation on this; LB1712 concluded 'Fine Sand' and LB1715, (who only used laser diffraction) concluded 'Muddy Sand'. Pre- analysis the majority of laboratories characterised the sample as muddy sand with some gravel, with the exception of LB1701 (Slightly shelly, muddy sand) and LB1715 (Muddy sand).

It is essential that analytical methods, including pre-treatment, are stated when reporting or attempting to compare results. The situation is further complicated by the fact that the difference between the techniques and the effects of the pre-treatment also varies with the nature of the sediment sample. As demonstrated in these and previous PS exercises, possible variations in equipment and methods can result in highly variable data. In order to eliminate as much variation as possible the NMBAQC's Best Practice Guide was devised for use in Scheme Year 17. Although most laboratories used the methods detailed in this document, a few labs still used in-house methodologies. All laboratories involved in CSEMP sample analysis used the NMBAQC PSA SOP for supporting biological data.

2.2.4 Application of NMBAQC Scheme Standards

One of the key roles of the Particle Size Analysis component of the NMBAQC Scheme is to assess the reliability of data collected as part of the Clean Seas Environment Monitoring Programme (CSEMP; formerly UK NMMP). With this aim performance target standards were defined for certain Scheme modules and applied in Scheme year three (1996/97). These standards were the subject of a review in 2001 (Unicomarine, 2001) and were altered in Scheme year eight; each performance standard is described in detail in the Description of the Scheme Standards for the Particle Size Analysis Component document. In previous years laboratories meeting or exceeding the required standard for a given exercise would be considered to have performed satisfactorily for that particular exercise. A flag indicating a 'Pass' or 'Fail' would be assigned to each laboratory for each of the exercises concerned. As the Pass/fail criteria is under review for the PS exercises in Scheme Year 17, a 'Pass' or 'Fail' flag will not be assigned to each lab for these particular exercises.

2.2.4.1 Laboratory Performance

Z-scores and cluster dendrogram figures are presented in each of the PS exercise reports, however these are only for illustration purposes. The investigations into new pass/fail standards are still underway. Pass/fail criteria will be introduced when sufficient data are collected using the new analysis guidance method.

3. Conclusions and Recommendations

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

- 1. Laboratories should endeavour to report their PS results in the requested format, e.g. at half phi intervals. This would enable the direct comparison of data from all participants and simplify the creation of cumulative curve figures. A modified workbook has been designed for use in Scheme Year 18 to enable labs to provide data in a comparable format. Participants should review their data prior to submission; zeros should only appear in submitted data where no material was present; dashes, '-', should appear where analysis has not been conducted.
- 2. Laboratories involved in CSEMP data submission should endeavour to return data on ALL necessary components of the Scheme in the format requested. This will be required to allow the setting of performance "flags". Non-return of data will result in assignment of a "Fail" flag. For CSEMP laboratories this deemed "Fail" for no submitted data is to be perceived as far worse than a participatory "Fail" flag.
- 3. Particle size exercises (PS) over the past sixteen years have shown differences in the results obtained by different techniques (laser and sieve / pipette), in-house methods (e.g. pre-treatment) and also differences between equipment (e.g. Malvern Mastersizer 2000, Mastersizer X and Coulter LS230 lasers). PS data indicates that the variance between laser and sieve results is further emphasised by certain sediments characteristics. The overall range of these variances needs to be determined if combining data sets derived from differing methods. The NMBAQC's Best Practice Guide has been developed for use in Scheme Year 17; this has helped to reduce the amount of variation between methods. It is essential that particle size data are presented with a clear description of the method of analysis and equipment used.
- 4. An improved learning structure to the Scheme through detailed individual exercise reports has been successfully implemented and was continued in this Scheme year. For the PS exercises, detailed results have been forwarded to each participating laboratory as soon after the exercise deadlines as practicable. Participants that submit significantly incorrect data are contacted immediately to ensure that in-house checks can be implemented to ensure future quality assurance. The PS36, PS37, PS38 and PS39 reports included the data submission sheets received from all participants as an appendix; Participants are encouraged to review their exercise reports and provide feedback concerning content and format wherever appropriate.

5. The current NMBAQC Scheme standards for PSA are under review. The alternative use of z-scores for each phi-interval, trialled in Scheme Year 17 appears inappropriate for such a low number of data returns where two erroneous results can significantly alter the pass / fail criteria. The z-score method also assumes that the majority of respondents are correct and raised genuine concerns regarding technique and method bias. Alternative flagging criteria will be trialled for use in Scheme Year 18 (2011/12).

4. References

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