The National Marine Biological Analytical Quality Control Scheme

Particle Size Analysis Component Report from the Contractor Scheme Operation – Year 15 2008/09

David Hall
davidhall@unicomarine.com

March 2010

Unicomarine Ltd.
Head Office
7 Diamond Centre
Works Road
Letchworth
Hertfordshire
SG6 1LW
www.unicomarine.com

PARTICLE SIZE ANALYSIS COMPONENT REPORT FROM THE CONTRACTOR

SCHEME OPERATION – YEAR 15 – 2008/09

1. Introduction **2**

1.1 Summary of Performance *2*

2. Summary of PSA Component **3**

2.1 Introduction *3*
2.1.1 Logistics 3
2.1.2 Data returns 3
2.1.3 Confidentiality 3

*2.2 Particle Size Analysis (**PS**) Module* *3*
2.2.1 Description 3
2.2.2 Results 4
2.2.3 Discussion 6
2.2.4 Application of NMBAQC Scheme Standards 7

3. Conclusions and Recommendations **7**

4. References **8**

Linked Documents (hyperlinked in this report)

Particle Size Results – PS32

Particle Size Results – PS33

Description of the Scheme Standards for the Particle Size Analysis Component

1. Introduction

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 fifteenth year of the Scheme (2008/09) followed the format of the fourteenth 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 / NMBAQC, 1995 & 1996).

Thirteen laboratories participated in the particle size analysis component of the NMBAQC Scheme (including one laboratory utilising replicated data). Seven laboratories were government laboratories; six were private consultancies. Over half of the participants (7) were responsible for CSEMP (Clean Seas Environment Monitoring Programme) 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 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 components of the Scheme, although this was not strictly enforced.

1.1 Summary of Performance

This report presents the findings of the Particle Size Analysis components for the fifteenth year of operation of the National Marine Biological Analytical Quality Control (NMBAQC) Scheme.

This component consisted of one module with two exercises:

- Analysis of two sediment samples for physical description (Particle Size module).

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

The **Particle Size exercises (PS)** were conducted as in the previous Scheme year. 'Pass/fail' criteria were applied 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 influence of in-house methodologies on the results returned for the PS32 exercise was clear in the two sets of *replicate* results produced by the benchmark laboratories. In most cases there was reasonably good agreement between participant laboratories for both PS exercises. The first particle size exercise of the Scheme year (PS32; sandy mud sample) received twelve data returns (including replicated data) that resulted in three 'fail' and fifty-seven 'pass' flags; two of these fails were the result of transcription errors. The second particle size exercise of the Scheme year (PS33; sand sample) received twelve data returns (including replicated data) that resulted in eleven 'fail' and forty-nine 'pass' flags; seven of these fails, produced by two participants, are likely to be the result their 'non-mainstream' processing methodology.

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 has received a 'Statement of Performance', which includes 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 / NMBAQC, 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 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. Each Scheme year fifteen participant was given a confidential LabCode in September 2008, these codes were randomly assigned. These 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 fifteen will be recorded as LB1504.

In this report all references to Laboratory Codes are the post-August 2008 codes (Scheme year fifteen), unless otherwise stated. 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 Unicomarine administering these three components).

2.2 Particle Size Analysis (**PS**) Module

2.2.1 Description

This component examined the production of derived statistics from the particle size analysis of replicate sediment samples. Two samples of sediment, one coarse the other finer, were distributed in 2008/09. Both sets of replicate samples, PS32 and PS33, were derived from natural marine sediments; they were prepared as described below. In each case a random subsample of the prepared replicates were divided for laser diffraction analysis using two differing instruments at separate laboratories to ensure sample replicate consistency and illustrate any potential variations between the laser instruments. For PS32 replicates were analysed using a Malvern laser (Mastersizer 2000) and a Coulter laser (LS230). PS33 *replicates* were analysed using Malvern Mastersizer 2000 and Malvern Mastersizer X instruments.

2.2.1.1 Preparation of the Samples

For both PS circulations sediment was collected from a natural marine environment (River Orwell for PS32; Wells-next-the-Sea for PS33). This material was returned to the laboratory and coarse sieved (2 mm) to remove gravel, shell and large faunal content. A minimum of 30 litres of visually similar sediment was collected for each circulation. Following sieving, the sediment for each PS circulation was well mixed in a large tray and allowed to settle for a week. Each 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 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. For PS32, half the *replicates* were analysed using a Malvern Mastersizer 2000 laser and half by a Coulter LS230 laser. For PS33, half the *replicates* were analysed using a Malvern Mastersizer 2000 laser and half by a Malvern Mastersizer X log bed 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 using their normal technique (either in-house or using a subcontractor) and to return basic statistics on the sample including %< 63µm, mean, median, sorting and skewness. A written description of the sediment characteristics was to be recorded (pre-processing and post-processing using the Folk Triangle) along with an indication of any peroxide treatment. Also requested was a breakdown of the particle size distribution of the sediment, to be expressed as a weight of sediment in half-phi (ϕ) intervals. **Eleven weeks** were allowed for the analysis of each PS sample (PS32 and PS33).

2.2.2 Results

2.2.2.1 General comments

The exercises in 2008/09 were undertaken by thirteen laboratories. One of the laboratories did not submit returns for either of the exercises; this is indicated in the tables by a dash (-).

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 are for 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 PS32, twelve out of thirteen participating laboratories returned data (including laboratories with grouped results); one laboratory did not provide data or provide notification of abstention. For PS33, twelve out of the thirteen participating laboratories returned data; one laboratory did not provide data or provided notification of abstention. Detailed results for each exercise have been reported to the participating laboratories (PS32 and PS33); additional comments are added below.

2.2.2.2 Analysis of sample replicates

Replicate samples of the sediment used for the two PS distributions were analysed using two different laser diffraction instruments. *Replicates* have previously been examined by both laser and sieve/ pipette methods, however as the majority of laboratories are conducting analyses by laser diffraction the testing of *replicates* is now undertaken using two different laser instruments. For PS32, half the *replicates* were analysed using a Malvern Mastersizer 2000 laser and half by a Coulter LS230 laser. For PS33, half the *replicates* were analysed using a Malvern Mastersizer 2000 laser and half by a Malvern Mastersizer X log bed laser. *Replicate* analyses were performed by Plymouth University, Geography Department (Malvern Mastersizer 2000), Partrac (Coulter) and Martin Ryan Marine Science Institute (Malvern Mastersizer X, log bed).

Some clearly significant methodology differences were noted between the data sets supplied by the two laser instruments, however the seven PS32 *replicate* samples analysed by each instrument showed good

agreement. There was very good agreement between the *replicate* samples analysed using the Malvern Mastersizer 2000 laser; the Coulter LS230 laser results showed more variability (see Figure 1 in PS32 Report). The Malvern instrument classified the PS32 *replicate* samples as sandy mud samples; the Coulter classified them as mud samples. The shape of the cumulative distribution curves differed between the two laser instruments; this divergence is likely to be the result of processing errors associated with the Coulter analyses. The figures for %<63µm varied significantly between the two instruments with the Malvern instrument producing an average figure of 58.9% and the Coulter 100%. Consequently, the derived statistics differed significantly between the two instruments. Results for the individual *replicates* are provided in Table 1 and are displayed in Figure 1 (PS32 Report).

Sample PS33 comprised sandy sediment (average of 0.21% <63µm). The Malvern Mastersizer X results showed a relatively high degree of variation between the PS33 *replicate* samples; the Malvern Mastersizer 2000 showed no discernable variation between *replicate* samples. As with the PS32 results, potentially significant methodology differences were noted between the data sets supplied by the two laser instruments. Differing cumulative distribution curves were once again produced by the two instruments (Malvern Mastersizer 2000 and Malvern Mastersizer X). The divergence of replicates illustrated by the Mastersizer X results is likely to have been influenced by low number of subsample replicates analysed to produce the data set for each sample, *i.e.* natural variation within each PS sample has not been adequately 'averaged'. The Mastersizer 2000 instrument produced an average silt/clay content figure of 0.00%; this figure was 0.41% for the Mastersizer X data. Results for the individual *replicates* are provided in Table 1 and are displayed in Figure 1 (PS33 Report).

2.2.2.3 Results from participating laboratories

Summary statistics for the two PS circulations are presented in Table 1 in each individual exercise report (see PS32 Report and PS33 Report). After resolution of the differences in data format, the size distribution curves for each of the sediment samples were plotted and are presented in Figures 2 in each individual exercise report (see PS32 and PS33). Included in each of these Figures, for comparison, are the mean distribution curves for the *replicate* samples as obtained by Unicomarine Ltd. (using Malvern Mastersizer X and Coulter LS230 instruments in PS32; using Malvern Mastersizer 2000 and Mastersizer X in PS33), Figure 3 in the individual exercise reports show the z-scores for each of the derived statistics. The z-scores were calculated with outliers and replicated data removed from the mean estimations of each of the major derived statistics.

One laboratory, which normally sub-contract their particle size analysis to another laboratory (also participating), elected to utilise the results from this laboratory for PS32 and PS33; this laboratory's data are regarded as replicated data and are not included in the calculation of z-scores. This laboratory is indicated in the report Tables by an asterisk against their LabCode. Accordingly the results from the sub-contracting laboratory have been used in the Figures and Tables as appropriate. In Figures 2 and 3 (PS32 and PS33) only data from the sub-contracting laboratory are displayed, although it also applies to the contracting laboratory. For Table 1 in each report, which present the summary statistics, although the results are displayed for all participating laboratories the replicated data supplied by the centralised laboratory (sub-contractor) have been included only once in the calculation of mean values for each exercise. Performance flags (as discussed in Section 2.2.4: Application of NMBAQC Scheme standards) have been assigned to laboratories using replicated data in the same manner as for other laboratories.

2.2.2.3.1 Thirty-second distribution – PS32

Excluding the replicate testing data set obtained by Partrac (Coulter LS230; see 2.2.2.2 Analysis of sample replicates) that showed no material present other than silt / clay, there was reasonably good agreement for PS32 between the results from the analysis of *replicates* and those from the majority of participating laboratories. One laboratory (LB1509) recorded a mean figure of 8.9ϕ, which was opposed to the majority of results and was not reflected in their raw data. One laboratory (LB1501) provided a figure for Inclusive Graphic Skewness that was not mathematical possible and did not reflect their raw data. 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 62.0% to 89.6%, excluding data from the *replicate* analyses produced by Unicomarine Ltd.

2.2.2.3.2 Thirty-third distribution – PS33

There was generally good agreement for PS33 between the results from the analysis of *replicates* and those from the majority of participating laboratories (see Figure 2). One laboratory's data (LB1504) showed significantly higher proportion of fine material. Also the results from LB1501 and LB1508 were notable atypical and their cumulative curves were displaced to produce finer median values. All of the participants used a combination of sieving and laser diffraction to analyse the sample; the aforementioned LB1501 and LB1508 were the only participants to undertake laser diffraction for just the silt/clay fraction, *i.e.* all other participants used laser diffraction for to analyse particles smaller than either 1 or 2 mm. Table 1 shows the variation in data received from the participating laboratories. The derived statistic for %silt/clay ranged from 0.0% to 3.5%, excluding data from the *replicate* analyses produced by Unicomarine Ltd.

2.2.3 Discussion

The difference between the laser instruments employed for particle size *replicate* analysis (Malvern Mastersizer X, Mastersizer 2000 and Coulter LS230) was evident, however the differences observed in PS32 and PS33 are likely to have been more influenced by the differing in-house analysis methods (*e.g.* hydrogen peroxide pre-treatment to remove organic material and use of sodium hexametaphosphate as a chemical dispersant to separate) than laser instrumentation. Partrac's data (Coulter LS230) received for PS32 indicated no material less than 5phi (greater than coarse silt), hence this set of results are clearly atypical in the cumulative curve figure (Figures 1 and 2). Consequently the *replicate* data supplied by Partrac for PS32 failed all the NMBAQC Scheme z-score standards; this error also occurred with Partrac's analysis of PS28 *replicates.* PS28 and PS32 were both circulations of sandy mud, which suggests a methodological error associated with this sediment type that is yet to be resolved. NMBAQC Scheme standard methods are to be devised to minimise the potentially significant difference in PSA data observed by pre-treatment.

The sample distributed as PS32 appeared from an analysis of *replicates* by Malvern Mastersizer 2000 (Figure 1) to be very uniform and the results from participating laboratories (Figure 2) showed a general similarity in distribution curves, however the spread of %silt/clay results ranged from 62 to 89%. Figure 3 shows the z-scores for each of the major statistics supplied by the participating laboratories. Two laboratories (LB1501 and LB1509), despite producing a relatively typical cumulative curve, supplied erroneous data for their sample's Inclusive Graphic Skewness and mean particle size, respectively; these have been confirmed as transcription errors.

The samples distributed as PS33 appeared from an analysis of *replicates* (Figure 1) to be good replicates with little variance within the two sub-sets of samples (Malvern Mastersizer 2000 and Malvern Mastersizer X). Results from participating laboratories were relatively well grouped (Figure 2). Figure 3 shows the z-scores for each of the major statistics supplied by the participating laboratories. Seven of the eleven 'fail' flags assigned via the z-scores are likely to be the result of two laboratories following a differing processing methodology that can result in finer data; LB1501 and LB1508 conducted sieve analysis down to 63µm and utilised laser diffraction for just the silt/clay fraction. A standardisation of PSA methods will eradicate such 'fail' flags produced by majority rule.

Participating laboratories were asked to provide a visual description of the PS32 and PS33 samples prior to analysis. The results varied considerably and some were extremely descriptive (Table 1, final column, in PS32 and PS33). Participating laboratories were also instructed to describe the sediment using the Folk triangle after analysis. Data were provided by eleven laboratories for PS32 and twelve laboratories for PS33. Eight of the eleven laboratories, that submitted data using the Folk triangle, described PS32 as 'Sandy mud'; one recorded 'Mud'; One recorded 'Poorly sorted medium silt'; and one described '(g)sM'. Ten of the twelve laboratories, that submitted data using the Folk triangle, described PS33 as 'Sand'; one recorded 'Medium Sand'; and one laboratory recorded 'Well sorted medium sand'.

It is essential that analytical methods, including pre-treatment, are stated when reporting or attempting to compare results. The situation is complicated further 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 a detailed and prescriptive method for particle size analysis must be devised for the CSEMP sample analysis.

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. 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. It should be noted that, as in previous years, only the Scheme's OS and PS exercise have been used in 'flagging' for the purposes of assessing data for the CSEMP. As the Scheme progresses, additional exercises may be included. In the meantime, the remaining modules and exercises of the Scheme are considered of value as more general indicators of laboratory performance, or as training exercises.

If a participant failed to return results for the PS module resulted in the assignment of a "Fail" flag to the laboratory for each pass / fail assessment. The only exception to this approach has been in those instances where laboratories elected not to participate in a particular exercise.

2.2.4.1 *Laboratory Performance*

The z-scores and results in each of the two PS exercises are presented in Table 2 and Figure 3 in the PS32 and PS33 Reports. The assigned flags for each laboratory for each derived statistic are also given. Where no returns were made for an exercise this is indicated in the tables with a "-".

Application of the new PS exercise standards, introduced in Scheme year nine, (see Description of the Scheme Standards for the Particle Size Analysis Component) is shown in the PS32 and PS33 Particle Size Results reports (see Table 2 and Figure 3 in the PS32 and PS33 Reports). Table 2 shows the results for the PS32 exercise. One laboratory (LB1502) is deemed to have failed all criteria due to non-submission of data. All participating laboratories passed the standard for %< 63µm, median (ϕ) and sorting; two laboratories (LB1509 and LB1526) failed the standard for mean (ϕ); one laboratory (LB1501) failed to meet the standard for IGS (SKi). Nine of the twelve participating laboratories passed all standards (LB1503, LB1504, LB1505, LB1506, LB1507, LB1508, LB1510, LB1511 and LB1527).

Table 2 shows the results for the PS33 exercise. One laboratory (LB1502) is deemed to have failed all criteria due to non-submission of data. Two participating laboratories (LB1504 and LB1508) failed to meet the standard for %< 63µm; three laboratories (LB1501, LB1504 and LB1508) failed the standard for median (ϕ); three laboratories (LB1501, LB1504 and LB1508) failed to meet the standard for mean (ϕ); one laboratory (LB1527) failed to meet the standard for sorting; two laboratories (LB1501 and LB1508) failed to meet the standard for IGS (SKi). Eight of the twelve participating laboratories passed all standards (LB1503, LB1505, LB1506, LB1507, LB1509, LB1510, LB1511 and LB1526).

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. Participants should review their data prior to submission; several PS failures have been the direct result of faulty spreadsheet formulae and straightforward transcription errors.
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 fifteen 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. It is essential that particle size data should be presented with a clear description of the method of analysis and equipment used.
4. PS exercises have highlighted the need for a prescriptive method for laser analysis (including equipment specifications) for the analysis of CSEMP samples. Replicate samples analysed using the same broad technique can result in highly variable summary statistics. A particle size standard operating procedure is to be developed through the NMBAQC Scheme for the CSEMP. The final draft will accommodate consultation and feedback from all significant parties.
5. 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 PS33 report included the data submission sheets received from all participants as an appendix; this revised reporting structure will be continued for all future PS reports. Participants are encouraged to review their exercise reports and provide feedback concerning content and format wherever appropriate.
6. Accurate representation of PS circulated samples using laser analysis can only be achieved via analysing multiple subsamples of the material. Several subsamples should be prepared from the bulk sample and these in turn analysed several times by laser diffraction. The final PSA results should be an average of these analyses.
7. The current NMBAQC Scheme standards for PSA need to be reviewed. The use of z-scores is 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.

4. References

Folk, R.L. (1974) *The Petrology of Sedimentary Rocks.* Hemphill Publishing Co. Texas

Hall, D.J. (2007) *National Marine Biological Analytical Quality Control Scheme. Particle Size Results: PS32.* Report to the NMBAQC Scheme participants. Unicomarine Report NMBAQCps32, October 2008.

Hall, D.J. (2008) *National Marine Biological Analytical Quality Control Scheme. Particle Size Results: PS33.* Report to the NMBAQC Scheme participants. Unicomarine Report NMBAQCps33, April 2009.

Hall, D.J. (2010) *National Marine Biological Analytical Quality Control Scheme. Description of Scheme Standards for the Particle Size Analysis Component from Scheme Year 8 (2001/02) to Year 16 (2009/10).* Report to the NMBAQC Scheme participants. Unicomarine report NMBAQCpsa_stds, February 2010.

Unicomarine Ltd. / National Marine Biological AQC Committee (May 1995). *National Marine Biological Quality Control (AQC) Scheme. Final Report April 1994 – March 1995*, 33pp. Tables 1-22, Figs.1-16.

Unicomarine Ltd. / National Marine Biological AQC Committee (June 1996) *National Marine Biological Quality Control Scheme. Final Report (Year 2),* 42pp.,Tables 1-17, Figs.1-16.

Unicomarine (2001) *National Marine Biological Analytical Quality Control Scheme. Own Sample Format and Standards Review: Current Problems and Proposed Solutions.* Report to the NMBAQC Committee. April 2001.